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Lockheed
ENVIRONMENTAL SYSTEMS & TECHNOLOGIES CO.
TECHNOLOGY APPLICATIONS DIVISION

PLUTONIUM IN SOILS TREATABILITY STUDIES ROCKY FLATS PLANT OPERABLE UNIT 2

MARCH 31, 1994

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EXECUTIVE SUMMARY

The Plutonium in Soils Treatability Study contains results of bench and laboratory scale tests which were performed on soils obtained from the Rocky Flats Plant site east of the 903 Pad. The treatability studies were performed under the guidance of the Plutonium in Soils Treatability Studies Work Plan. The primary objective of the Plutonium in Soils Treatability Study was to evaluate the ability of the TRUclean process to reduce activity levels of plutonium, gross alpha, and gross beta in Rocky Flats soils below established criteria.

The treatability studies consisted of three stages: Soil Characterization studies, Phase 1 treatability testing, and Phase 2 treatability testing. The Soil Characterization stage determined specific characteristics of the soil which provide information for the Phase 1 and Phase 2 tests. The Phase 1 tests were performed to define treatment sequence and optimum equipment settings for the Phase 2 tests. The Phase 2 tests consisted of four test runs using 90-110 kg of soil. Processing equipment was operated at the optimum settings for reducing activity levels in the soil.

The soil used in the treatability study was screened in the field to remove all gravel and cobbles larger than 5 centimeters (2 inches). This represented 21.1% of the soil mass. The soil shipped to the Lockheed laboratory had an activity of 76 pCi $^{239+240}$Pu/g.

Summarizing the characterization results, the soil was found to have a slightly alkaline pH of 7.61, a loose-state bulk density of 1.02 g/cm^3, and a weight percent of the less-than 45 micron fraction (clays and most silts) of 27.4% (for received soil). Plutonium activity ranged from less-than 1.0 pCi $^{239+240}$Pu/g in several gravel size fractions to 143 pCi $^{239+240}$Pu/g in the less-than 45 micron fraction. Over 65% of the total plutonium activity resided with the less-than 45 micron soil.

Decomposed and undecomposed natural organic matter, comprised primarily of grass species, was evident throughout every size fraction. In both the characterization tests and the Phase 1 and 2 treatability tests, all separated organic matter was found to contain ^{241}Am activity. The nature of the association of the activity to the organic matter was not investigated.

Lab scale testing during Phase 1 demonstrated the ability of autogenous grinding and attrition scrubbing techniques to remove surface deposited americium and plutonium from coarse grained material. Autogenous grinding for 5 to 10 minutes reduced starting activity levels of 0.5 pCi ^{241}Am/g in the >6.33 mm fraction down to a range of 0.09 to 0.15 pCi ^{241}Am/g. Attrition scrubbing tests succeeded in reducing starting activity levels of 1.5 down to 0.2 pCi ^{241}Am/g for the 0.85 mm fraction, of 10.1 down to 0.5 pCi ^{241}Am/g for the 0.30 mm fraction, and of 9.5 down to 2.4 pCi ^{241}Am/g for the 0.15 mm fraction.

Testing of bismuth and magnetite surrogates on the gravity separation portion of the TRUclean system permitted optimization of processing variables and equipment sequences, resulting in recoveries of 70 to 80%.

In Phase 2, processing the soils through dry screening and trommel scrubbing resulted in 23.8% of the soil meeting the 0.9 pCi $^{239+240}$Pu/g cleanup criteria. The soil processed through the gravity separation system did not produce any concentrates which had elevated levels of plutonium. The concentrates did, however, have elevated levels of naturally occurring radioactive materials.

The mineral jig was used in the gravity system to concentrate dense particles having diameters of about 50 microns or larger. Process streams from the mineral jig contained residual activity levels of 6.0 to 15.4 pCi $^{239+240}$Pu/g, representing 27% of the feed volume. No evidence of individual plutonium oxide particles was found in mineral jig concentrates. Process fines (soil material less-than about 75 micron diameter), accounting for 26.1% of the whole soil, had elevated activity levels of from 76 to 377 pCi $^{239+240}$Pu/g, and accounted for more than 94% of the activity found in the feed soil. Organic matter reporting out in process streams was found to elevate sample activity levels.

Recommendations for further study are provided, based upon the findings of this work.

ACRONYM LIST

Am	Americium
AP	Analytical Procedure
ASME	American Society of Mechanical Engineers
ASTM	American Society for Testing and Materials
ARAR	Applicable or Relevant and Appropriate Requirements
C	Celsius
$CaCl_2$	Calcium Chloride
CAR	Corrective Action Report
CLP	Contract Laboratory Procedures
cm	Centimeter
cm^3	Cubic Centimeter
DOE	Department of Energy
DOT	Department of Transportation
EPA	Environmental Protection Agency
FSP	Field Sampling Plan
g	Gram
G	Gravitation Constant
HEPA	High Efficiency Particulate Adsorber
HGMS	High Gradient Magnetic Separation
ID	Integrated Demonstration
in	Inches
ITC	International Technology Corporation
keV	Kiloelectron Volts
kg	Kilogram
kPa	KiloPascal
L	Liter
LAB	Laboratory
LAL	Lockheed Analytical Labs
lbs	Pounds
LESAT	Lockheed Environmental Systems and Technologies Company
LES&T-CS	Lockheed Characterization Sample
LSCOPP	Land Surface Cleanup of Plutonium Project
M	Molar

ACRONYM LIST
(continued)

MCA	Multi Channel Analyzer
M.D.	Minimum Detectable
min	Minute
mL	Milliliters
mm	Millimeter
NaI	Sodium Iodide
NaOH	Sodium Hydroxide
NCR	Non Conformance Report
NIST	National Institute of Standards and Technology
NORM	Naturally Occurring Radioactive Materials
NTS	Nevada Test Site
OTD	Office of Technology Development
OU2	Operable Unit 2
pCi	Picocurie
Pu	Plutonium
QA	Quality Assurance
QAPP	Quality Assurance Program Plan
QP	Quality Procedure
RFP	Rocky Flats Plant
RI/FS	Remedial Investigation/Feasibility Study
ROI	Region of Interest
RPM	Revolutions Per Minute
SN	Serial Number
SOP	Standard Operating Procedure
SRCC	Standard Radiological Counting Container
SVOC	Semi-Volatile Organic Compounds
TAD	Technology Applications Division
TAL	Target Analyte List
TSP	(Final) Treatability Study Plan
VOC	Volatile Organic Compounds

1.0 INTRODUCTION

This report provides a summary of treatability studies using the TRUclean process on soil, which contains plutonium, collected several hundred meters east of the 903 Pad area at the Rocky Flats Plant (RFP). The TRUclean process was selected as a commercially available technology in the treatment of soils containing radioactive materials. Data obtained during these tests are presented in this report and will be utilized in the future evaluation of treatment alternatives during Feasibility Studies at various operable units at RFP.

In 1991, the DOE Office of Technology Development (OTD) initiated the Integrated Demonstration (ID) program to characterize and evaluate treatment alternatives for soils containing non-native radionuclides (specifically plutonium), and potential remediation activities. This program combined efforts from different DOE facilities which have similar situations.

Due to a schedule commitment of the Interagency Agreement, EG&G RFP initiated a separate treatability study independent of the ID effort. The study was performed in parallel with DOE OTD to perform treatability studies on soils containing plutonium.

In August of 1991, the Final Treatability Study Plan (TSP) was developed to meet the requirements of the Final Interagency Agreement (Article XI). This TSP identifies candidate technologies for treatment of different contaminants at RFP for use in corrective/remedial actions through the screening/selection process. The TRUclean process was among the technologies selected for further evaluation through bench and laboratory testing for soils containing plutonium at RFP (specifically the OU2 Area).

In February 1993, the final Plutonium in Soils Treatability Studies Work Plan for the TRUclean process was approved by the EPA Region VIII and Colorado Department of Health for implementation. Findings of these studies are presented in this report.

1.1 Site Description

1.1.1 Site Name and Location

The Rocky Flats Plant (RFP) is a U.S. Department of Energy facility located approximately 16 miles northwest of Denver, Colorado. The plant site is located on a 6550 acre reservation of federally-owned land in northern Jefferson County. The majority of the plant buildings are located within a 400 acre area referred to as the RFP security area. The balance of the reservation provides a buffer zone around the RFP security area.

1.1.2 History of Operations

The facilities and reservation are currently managed by EG&G Rocky Flats, Inc. for the Department of Energy. Previous operations of the facilities were managed by Rockwell International and Dow Chemical for the Department of Energy.

During the early 60's several drums of cutting oil containing plutonium were stored in the area now containing the 903 Pad. These drums developed leaks and the contents spread onto nearby soils. Wind dispersion in the following years resulted in the plutonium spreading to adjacent areas. The OU2 area, which is the subject of this treatability study, includes the 903 Pad, Mound, and East Trench areas.

1.1.3 Prior Removal and Remediation Activities

During 1967 through 1976, the 903 Drum Storage Area and mound underwent partial remediation efforts to remove soils with high plutonium activities, alter surface drainage, and stabilize soil with dust suppressants and area capping. These actions were performed to address interim remediation for protection of human health and the environment while long-term solutions were evaluated.

1.2 Waste Stream Description

For the purposes of this work, the waste stream is identified as being the in-situ soil containing plutonium released at the 903 Pad.

1.2.1 Waste Matrices

The matrix (soil) containing the target material (plutonium) which was sampled is one of the more common soil classes present at OU2 (U.S. DOE, 1992). The soils in the area were described and mapped by the U.S. Department of Agriculture.

A profile description of the soil sampled for this study was not available at the time of this report.

1.2.2 Pollutants/Chemicals

The chemicals/isotopes studied in this work are the major isotopes of plutonium released at the 903 Pad (^{238}Pu and ^{239,240}Pu), and the daughter product, ^{241}Am, of ^{241}Pu. The isotope ^{241}Am was chosen as an indicator for the plutonium isotopes, gross alpha, and gross beta. The isotopes ^{239,240}Pu were then calculated from the established ratio of ^{239,240}Pu to ^{241}Am and used as the single indicator for the suite of radionuclides and gross alpha/gross beta.

Starting and processed samples were also analyzed for CLP SVOCs and VOCs, and TAL metals. These analyses were adjunct to the objectives of this work. Results of these analyses are contained in Appendix A.

1.3 Treatment Technology Description

The TRUclean process consists of a set of modular-like units (equipment) which together perform particle-surface scrubbing (liberation) and gravity/size/shape differentiation (separation).

1.3.1 Treatment Processes and Scale

The processing train for the TRUclean process involves an initial liberation of the target material (plutonium on fine grain particles) from the surfaces of coarse grain particles. The liberation process is by physical means and only involves chemicals as needed for pH adjustment of the bathing solution or as surface-active agents which aid in disrupting the attachment of the fines to the coarse material.

Two kinds of liberation processes were used for the configuration of TRUclean process used in this work. The first process involved the use of autogenous grinding in a trommel, the abrasive action resulting from the collision of very coarse gravel particles upon each other in a slurry. The autogenous grinding results in the surfaces being abraded clean of attached particles and coatings.

The second liberation process also involves the abrading action of particles upon each other, but uses fine gravels and sands in a slurry of a predetermined solids concentration. The particles are driven towards one another by opposing pitch impellers in a double cell attrition scrubber. This method also results in the "scrubbing" of particle surfaces to remove attached particles and coatings.

Three categories of separation processes were used in the TRUclean configuration applied in this project. The first category involves separation based upon size, with previously scrubbed, more coarse particles being separated off at various stages into cleaned, or lower activity, streams. As expected, plutonium deposited in soils at the 903 Pad generally concentrates in the finer material. The size separation of higher plutonium activity fines from lower plutonium activity coarse particles presents one means of an overall volume reduction of the starting soil.

The second category of separation processes applied to this work involved the separation of particles based upon density differences. Plutonium dioxide particles generally have densities near 11 g/cm^3, well above that for most native soil minerals (around 2.6 g/cm^3). Processes capitalizing on differences of initial falling velocities, solvent/particle density differences, or differences in response to elevated "G" (gravity) forces were applied to determine the separation efficiency of gravity processes.

The third category of separation processes studied used classification means of particle separation. Classification capitalizes on differences involving a combination of particle density, shape, and size, with generally smaller, more platy particles being separated off from more coarse, and more blocky-shaped or more dense particles.

Initial settings and optimal conditions and rates were determined using lab bench top equipment. Once optimized, larger lab-scale equipment was assembled for actual processing runs. At the lab scale, minor adjustments in residence times, flow rates, slurry densities, and equipment operating speeds were made to further optimize plutonium liberation and separation.

1.3.2 Operating Features

The operating features of the TRUclean process consist of a series of mineral processing equipment assembled together, with each unit optimized for its particular purpose, and the overall assembly optimized for processing runs.

The system begins by applying the liberation process of autogenous grinding in a trommel. The trommel is a rotating drum whose turning action causes the abrasive collision of coarse gravel particles crashing into one another. The trommel can be adjusted for rotational speed and angle of drum pitch, which determines residence time for a particular particle.

At the lower half of the trommel, a large diameter opening screen is attached. The separation process begins here as trommeled slurries wash over the screen. Fine material (sands, silts, clays, and fine gravel) are washed through the screen and collect in a chute for further processing. Cleaned coarse gravel reports out through a separate chute as a clean stream. During operation, a fine stream of water is sprayed from a pipe inserted longitudinally through the trommel. Screens can be changed to provide larger or smaller diameter openings.

The next stage of TRUclean processing is also a liberation step and involves a two cell attrition scrubber. Adjustments to the scrubber are slurry density, residence time, and impeller speed. These variables can be adjusted to provide greater abrasive action on fine gravels and sands.

Following the scrubbing of particles, the process stream reports out to a vibrating screen. The screen further separates scrubbed gravels from finer material. Screen opening size and clean water inflow can be adjusted to better effect the washing and removal of coarser grains.

The fines process stream then reports to the mineral jig, where separation based upon gravity, as initial settling velocities, takes place. As the process stream flows horizontally across the bed of steel shot, a pulse of water is pushed vertically out of the bed. More dense particles initially fall more rapidly towards the bed, whereas less dense particles are carried across both beds and out through a discharge chute. Due to the continuous pulsation of the beds, terminal settling velocities are never reached, thereby capitalizing on density differences which determine initial settling velocities. The more dense particles are worked through the beds and into the hutches. Slurry density, bedding thickness, hutch water flow, and stroke travel length and speed can be varied to optimize separations.

Material removed through the hutches reports to equipment which further separates heavy particles from less dense particles. A Wilfley gravity shaker table classifies particles based upon sizes and densities. Feed rate and density, wash water volume, side and longitudinal tilt, and reciprocating stroke length and frequency are all adjustments which can be varied to optimize separation.

The fines which are outflow from across the mineral jig are further processed in a spiral classifier. The spiral classifier effects separation based principally upon particle size, with cleaner, larger particles reporting out as underflow and finer particles reporting out as overflow. Auger speed, settling pool depth and surface area, feed rate and feed density are adjustable.

Overflow from the spiral classifier is further processed by first being filtered through a screen to remove larger sized organics. Overflow is then pumped into a thickener tank, where fine silts and clays can settle out. Though not used in this work, flocculating agents can be added into the thickener to further aid in the settling of fines.

Thickener underflow (concentrated fines) reports out to a centrifugal concentrator in which further separation based upon density differences occurs. Feed rate and density are adjusted for optimization. Centrifugal tail is further processed in a hydrocyclone to obtain a size cut down to around 10 microns. Feed density,

orifice shapes and sizes, and inlet pressure are adjustments which affect hydrocyclone separations.

1.4 Previous Treatability Studies at the Site

During 1987, the Department of Energy (DOE) sponsored a program to evaluate methods of reducing the volume of soils containing plutonium at several DOE sites. RFP participated in this program and sent soils to the Nevada Test Site (NTS) where testing was conducted. These tests were successful in reducing the levels of plutonium in soil and in concentrating the activity in the fine size fraction. High density materials collected as mineral jig concentrate (AWC, 1987).

The tests performed in 1987 did not provide adequate information to draw definite conclusions to support evaluation of the treatment alternatives during Feasibility Studies for radionuclides in soil.

Previous investigations in 1987 indicated that the TRUclean process could reduce plutonium activity in Rocky Flats soils down to levels approaching the current proposed performance criteria of 0.9 pCi $^{239+240}$Pu/g of soil (AWC, 1987). The treatability tests used in this study modified the feed preparation methods used in the original 1987 tests.

2.0 Conclusions and Recommendations

2.1 Conclusions

A summary of the Plutonium in Soils Treatability Study, conclusions drawn from the results, and an evaluation of the performance in terms of RI/FS evaluation criteria are discussed in the following sections.

2.1.1 Project Summary

Soil used for the treatability test was obtained by EG&G Rocky Flats using an approved sampling plan. This soil was screened in the field to remove all rocks larger than 5 centimeters (2 inches). Following collection, the samples were shipped to Lockheed's Las Vegas laboratory for characterization and the treatability study. After receipt at the laboratory, the samples were homogenized to provide a uniformly mixed sample for use in the characterization and treatability study.

During the Phase 1 characterization study, sample aliquots were analyzed for bulk density, percent air-dry moisture, pH, volatile and semi-volatile organics, TAL metals, gross alpha and gross beta, and activity in density separations which used heavy liquids. A sample was also subjected to dry and wet sieving using 15 sieves. This sieving allowed the evaluation of mass and activity distribution by particle size.

The Phase 1 treatability testing evaluated and optimized process variables and equipment settings which would be used in the Phase 2 treatability tests. The scrubbing time in the trommel was evaluated using a laboratory size ball mill to determine residual activity as a function of tumbling time. The laboratory scale attrition scrubber, containing between 500 and 1000 grams of sample, was used to evaluate and optimize scrubbing times for the larger scale attrition scrubber

used in the Phase 2 tests. The mineral jig and spiral classifier settings were optimized using surrogate material to verify that high recoveries would take place if plutonium existed in the recovery range of the mineral jig.

The Phase 2 treatability test used four (4) test runs to evaluate the effectiveness of size, shape, and gravity separation to remove plutonium from Rocky Flats soils. The first test run was used to stabilize equipment and establish a process heel in the attrition scrubber. The remaining three runs were performed to provide required data for the three runs required by the test plan. The test runs were subdivided to allow optimum control of process parameters and data collection. These tests included:

1. Initial dry vibratory screening of homogenized feed.

2. Wet trommel scrubbing and screening of >6.3 mm diameter soils.

3. Gravity separation of <6.3 mm diameter soils following attrition scrubbing.

4. Gravity separation of the fine soils collected in the thickener followed by size separation through a hydrocyclone.

All process streams were evaluated for mass and activity to determine recoveries. Process residues were dried to remove moisture and individually packaged for return to Rocky Flats for archive storage in early 1994.

2.1.2 Final Conclusions

The TRUclean process was successful in meeting the proposed treatability study plutonium performance goal of 0.9 pCi ^{239,240}Pu/g soil for the 6.3 mm (1/4") to 50.8 mm (2") particle size soil fraction (Table 2.1, >6.3 mm wet trommel). This represented 23.8% of the whole soil mass. ^{239,240}Pu was used as a single indicator species for the other proposed radioactive performance goals for gross alpha (5 pCi/g) and gross beta (50 pCi/g). All other process streams listed in Table 2.1 were above the ^{239,240}Pu performance criteria.

Trommel scrubbing (autogenous grinding) was the process found to be effective in meeting the ^{239,240}Pu cleanup criteria. The success of trommel scrubbing was due to the abrasive grinding action of gravel particles colliding with one another in the rotating drum. This abrasive action resulted in the breaking off (liberation) of particle-surface coatings and attached very-fine particles which carried the majority of plutonium for this fraction.

The success in meeting the proposed performance criteria for ^{239,240}Pu for the 6.3 to 50.8 mm fraction, and the less successful lowering of overall plutonium activity levels in other gravel and sand size fractions (Table 2.2, compare wet sieve fraction activities to Phase 1 and 2 fraction activities) was influenced, in large part, by what is stipulated to be the forms in which plutonium exist in these soils. Indirect evidence collected throughout the project indicates that all plutonium exists in either particle or molecular sizes, all less than a 5 to 10 micron diameter range.

Table 2.1 Results for Integrated TRUclean System

Process/ Waste Stream	Percent Mass		^{239,240}Pu Activity (pCi/g)	Standard Deviation, Population pCi/g	Standard Deviation, Counting pCi/g	Percent Activity (<50.8 mm)	
Field Screened (>50.8 mm)	21.1		?	----------	----------	----------	
Dry Screened Organics (>6.3 mm)	0.38		87.5	±49.1	±11.31, 11.58	0.5	
Wet Trommel (>6.3 mm)	23.8		0.86	----------	±0.669	0.3	
Attrition Scrub & Wet Screened (>4.8 mm)	1.02		15.4	±9.04	±2.10, 2.19	0.2	
Hutch 1, Table Tails	11.4		6.54	±2.97	±2.069, 2.152	1.1	
Hutch 1, Table Concentrate	1.1		<M.D	----------	±0.99	<M.D	
Hutch 2, Table Tails	1.72		11.0	±1.55	±2.54, 2.55	0.3	
Hutch 2, Table Concentrate	0.58		8.47	----------	±3.56	0.1	
Spiral Classifier Organics	0.52		157.0	±6.27	±10.6, 13.6	1.2	
Spiral Classifier Underflow	12.2		9.08	±3.14	±1.61, 1.71	1.7	
Centrifugal Concentrator, Concentrate	9.8	} 26.1%	76.1	--------	±3.963	11.2	} 94.6%
Hydrocyclone Underflow (104 kPa)	9.7		316.0	--------	±20.82	46.0	
Hydrocyclone Overflow (104 kPa)	6.6		377.0	----------	±28.18	37.4	

Notes: Where given, the Population Standard Deviation was derived from duplicate samples, each of which had a Counting Standard Deviation associated with the value. The Counting Standard Deviation was a product of gamma spectroscopic analysis and resulted from a series of counting cycles for the period of analysis.

Feed soil averaged 76 pCi^{239,240}Pu/g; soil going into the mineral jig portion of the system averaged 91.3 pCi^{239,240}Pu/g.

Table 2.2 Selected Compiled Results (pCi ^{239,240}Pu/g)

Size Ranges	Wet Sieve Activity	Phase 1 *Autogenous* Grinding	Phase 1 *Attrition* Scrubbing	Phase 2 *Trommel* Testing	Phase 2 Classifier Underflow, Sieved
6.3-51mm	3.11	0.86	----------	0.86 - 4.72	----------
0.15-6.3mm	29.5	----------	4.26	----------	5.59

The strongest evidence for concluding that the nature of the forms of plutonium in these soils hinders further separation of plutonium from the less-than 6.3 mm fraction are the findings that no gravity separation or classification process concentrated plutonium particles into a smaller volume of soil (Table 2.1). Elevation of activity levels in process fines was accomplished solely due to the removal from gravels and sands of surface-bound coatings and grains which themselves held plutonium by means of adsorption, precipitation, or co-precipitation.

In applications involving sand size (>50 microns) target particles, process run hutch concentrate products will typically contain activity levels higher than the starting activity level of unprocessed soil, due to the concentration of the more dense (11 g/cm^3) discrete plutonium oxide. Consistently though, hutch products were lower in plutonium activity levels by an order of magnitude or more (i.e., 6.54 to 11.0 pCi^{239,240}Pu/g, Figure 2.1) than the soil (91.3 pCi^{239,240}Pu/g) going into the mineral jig portion of the TRUclean system. Hutch products were found, however, to have elevated levels of NORM bearing minerals, denser than common silicate minerals. The concentration of NORM bearing minerals thereby verifies that the operational state of the mineral jig had been optimized. Hutch products further processed on a Wilfley shaker table gave an additional concentration of NORM, but no concentration of plutonium.

Results of post-thickener process runs on thickener product also provided strong evidence that the very small particle diameters of plutonium oxides directly impacted the inability for fines-separation equipment to concentrate plutonium.

Fine grained solids (<75 micron diameter), concentrated in the thickener, were subjected to further separation processes by use of a centrifugal concentrator at rotational speeds in excess of 300 Gs, and by use of a hydrocycle operated at inlet pressures of 104 kPa to 414 kPa. The results (Table 2.1) again show that a concentration of plutonium-bearing particles above 5.0 to 10 micron diameter size (the lower effective range for the hydrocyclone) does not occur, most likely due to the lack of any plutonium oxide particles above that range for this soil. Over 83% of the activity was present in the soil fraction tested by hydrocyclone, representing 16.3% of the whole soil mass.

The inability of TRUclean separation processes to concentrate plutonium oxide, whereas NORM bearing minerals were concentrating into smaller volumes, and the lack of identifying any discrete plutonium particles at all, led to the conclusion that all plutonium dioxide existed as particles or molecules less than the 5 to 10 micron split range for the hydrocyclone. Gravity separation and classification

processes were unsuccessful because the particle-diameter material which the equipment was designed for was not present in the test soil. Plutonium resided in the fine material (less than 75 micron diameter), giving various fractions activity levels ranging from 76 to 377 pCi ^{239,240}Pu/g (Table 2.1). This accounted for over 94% of the activity in the test soil, and 26.1% of the whole soil mass. A total activity balance for the integrated results (6659 pCi^{239,240}Pu/100g) was within 12.4% of the feed soil activity (7600 pCi^{239,240}Pu/100g, calculated from 76 pCi^{239,240}Pu/g).

Another conclusion to be drawn from the results is that americium (and by inference, plutonium) activity is associated with decomposed and undecomposed organic matter. In every instance, for both the characterization phase and the treatability phases, all samples analyzed which contained some, or nearly all, organic matter was found to have elevated activity levels (i.e., 87.5 pCi^{238,240}Pu/g soil, 6.3 mm dry screened organics, Table 2.1). Samples containing undecomposed organic matter, washed free of inorganic solids, also had elevated activity levels.

The soil processes (mechanisms) leading to the association of americium to organic matter could not be identified by the limited characterization work performed in this project and was not an objective addressed by the work plan. The removal of organic matter prior to processing or analysis was found, however, to benefit in lowering the overall activity of the samples removed for analysis (Table 2.2, Phase 2 classifier underflow, sieved). It was concluded, therefore, that any full scale processing system set up for treatment of the 903 Pad area soils should address the organic matter activity content.

Finally, with the removal of 21.1% of the soil mass in the form of greater than 5 centimeter gravels and cobbles (Table 2.1), it was concluded that trommel treatment of this mass, along with trommel treatment of the 6.3 mm to 51 mm gravel will result in an overall cleanup of 44.9% (by mass) of the soil on site. An additional 25.3% (Hutch 1 concentrate and tails, Hutch 2 concentrate, and classifier underflow) of the remaining soil can be scrubbed to within 1 order of magnitude of the proposed performance criteria of 0.9 pCi ^{239,240}Pu/g. If further treatment by scrubbing were successful, a combined total of 70.2% of the original soil mass might meet this goal.

2.1.3 RI/FS Evaluation Criteria

Evaluation of the results of this treatability study against the first seven of the nine identified Remedial Investigation/Feasibility Study (RI/FS) evaluation criteria (EPA, 1992) can be discussed in terms of the processes and equipment employed and the overall volume reduction achieved and anticipated.

Threshold Criteria

The first of the two threshold criteria, overall protection of human health and the environment, can be achieved based upon an achieved mass reduction of 23.8% and a combined achieved and assumed mass reduction total of 44.9% (including the >5 cm material removed in the field), using the 0.9 pCi ^{239,240}Pu/g

performance goal. Further treatment of the remaining 55.1% of the mass by other means or off-site disposal at a licensed facility would be required in order that the threshold criteria, as measured by the plutonium performance goal, be met.

Compliance with applicable or relevant and appropriate requirements (ARARs) can be achieved for the 23.8% mass meeting the 0.9 $pCi^{239, 240}Pu/g$ performance goal, and can be assumed met by the additional 21.1% field reject material (>51 mm). Location specific ARARs placed on cleanup activities will need to consider issues such as dust generation during excavation and system feeding.

Primary Balancing Criteria

The first of the five primary balancing criteria, long-term effectiveness and performance, will again be met for the total achieved and assumed 44.9% mass of soil, again using the 0.9 $pCi^{239, 240}Pu/g$ single indicator criteria. The TRUclean system, in the configuration adopted for this project, did not involve the use of any chemicals which may serve in the long term to mobilize toxic metals or additional plutonium from the treated material. At issue for post remediation residual risk, therefore, will be the 55.1% of soil not meeting the performance goal, this mass of which includes process residuals. Other action will be required.

The reduction of toxicity, mobility, and volume through treatment criterion can also be met for the combined achieved and assumed 44.9% mass by means of a reduction of the total volume of media. Indications are that additional attrition scrubbing of the 0.15 to 6.3 mm sand and gravel may contribute to surpassing a 50% threshold level.

The short-term effectiveness criterion can be met for both on-site workers and the community at large principally by methods which suppress the creation of fugitive dust during operations and protect workers against inhalation hazards. Release of process water from operations would not occur as the TRUclean process system configured for this project recycles the process water for reuse.

The implementability and cost criteria both can be addressed as similar configurations of the TRUclean process have been operated at different sites. The TRUclean process used was composed of off-the-shelf mining industry equipment in a configuration optimal for this work.

Modifying Criteria

The two modifying criteria, state acceptance, and community acceptance are beyond the scope of discussions of this report.

2.2 Recommendations

The following recommendations are offered to provide direction for future activities which will assist in further treatability studies for plutonium in soils at Rocky Flats.

Recommendation #1: Spatial Variability & Sample Lot Size

The distribution of plutonium outward from the 903 Pad may occur in isopleths, and in conjunction with the particle size distribution for the soils could contribute to the representativeness of the collected samples.

A determination of the spatial variability of the plutonium distribution, mapped as isopleths, and a consideration of field sampling under the concept of "fundamental error" (Pitard, 1989) should be evaluated.

As gravity based separation systems are sensitive to the particle size of the target material, different representative samples may respond differently to gravity separation systems. Soils closer to the 903 Pad may contain plutonium particles which can be separated out by gravity systems.

Recommendation #2: Homogeneity of Gamma Analyzed Samples

Some of the variability of activity levels in duplicate samples taken from gravel material could be attributed to uneven disbursement of fines over the surfaces of different sides of gravels and sands.

The activity in a sample should be distributed in the same manner as the standard against which it is measured, that being uniformly distributed throughout the sample. A method should be developed to prepare gravel and sand samples so that the activity in the samples is uniformly distributed, rather being located only at outer surfaces.

Recommendation #3: Removal of Submicron Plutonium by HGMS

Plutonium in the tested soils is indicated to exist as <10 micron diameter down to submicron particles.

An evaluation of the feasibility for using High Gradient Magnetic Separation (HGMS) to remove the submicron plutonium particles in this soil should be conducted to determine the effectiveness of HGMS treatment.

Recommendation #4: Activity and Treatability of >51mm Gravel and Cobbles

No activity level determination or treatability testing of the >51mm field rejected materials has been conducted.

The activity level of the rejected stone material should be determined. Additionally, if the activity levels are above the criteria, treatability of this fraction using dry screening, trommel scrubbing, and wet screening should be conducted to establish the residual activities.

Recommendation #5: Low Activity Soil Response to Treatment

Feed soils with lower activity levels should result in recovery of larger soil volumes meeting the established criteria.

Soil decontamination factors, as a function of the soil feed activity concentrations, should be verified to determine the viability of the process techniques used in this project. Using

the processes studied, soil recovery could be increased in soils with lower feed activity levels.

Recommendation #6: Development of Methods to Address Submicron Plutonium

Liberation and separation of plutonium particles in the 5 to 10 micron diameter range, and below, from like-sized aluminosilicate particles was not achieved in this project.

Studies for the development of methods which would liberate and separate out plutonium in the fine fractions (less-than 10 micron diameter) should be initiated, within the latitude of using chemical, microbial, electromagnetic, electrokinetic, or other physical treatments at Rocky Flats.

Recommendation #7: Association of Americium with Organic Matter

Consistently, in all phases of the project, no organic matter was found which was free of americium activity.

Association of americium, and by implication, plutonium, with organic matter does not appear to have been well established for soils at Rocky Flats by previous researchers. As all organic matter was found to be associated with some level of americium, a spectroscopic analysis of any americium/oxygen/carbon bonding environment should be conducted. This work could establish whether or not americium/plutonium is being taken up into plant tissues, and thus inaccessible to solutions, or held outside plant material (biosorbed), and thus accessible to solutions for liberation.

3.0 TREATABILITY STUDY APPROACH

3.1 Test Objectives and Rationale

The primary objective of the plutonium in soils treatability studies was to evaluate the ability of the TRUclean process to reduce the plutonium activity in RFP soil to an acceptable level.

Soil treatability study performance criteria for the RFP have been proposed based on human health, environmental risk assessment criteria and applicable state and federal requirements. These proposed performance criteria are 5 pCi/g for gross alpha, 50 pCi/g for gross beta, and 0.9 pCi/g for ^{239,240}Pu. For the purposes of this work, the sampling and analysis objectives were limited to address a single indicator (^{239,240}Pu), thereby increasing both the time and cost savings (EPA, 1992). The ratio of ^{239,240}Pu to ^{241}Am was established at the onset of this study and was used throughout the project.

Results of the TRUclean tests and all the data presented in this report are used to evaluate performance against the proposed ^{239,240}Pu criteria. Performance data is evaluated based on the residual activity (^{239,240}Pu) in the outlet streams of the TRUclean process and the degree of separation, that being the mass split between those soils meeting the residual activity cleanup criteria for ^{239,240}Pu, and those soils not meeting the cleanup criteria.

3.1.1 Scope of Work

The scope of work was specified in the Treatability Study Work Plan. A graphic representation of the tasks required by the work plan is shown in Figure 3.1. The work plan provided for homogenization of soils, characterization, Phase 1 testing, and Phase 2 testing. The Phase 1 treatability testing consisted of testing various laboratory scale equipment to determine the optimum sequence and settings of equipment and process variables. The primary objective of the Phase 2 treatability study was to evaluate the ability of a larger scale process to reduce the concentration of plutonium, gross alpha and gross beta in Rocky Flats soils.

3.1.2 Characterization Phase Objectives

The characterization phase objectives were to obtain data on soil properties which were relevant to the treatment phase of this project.

Homogenization

The objectives of homogenization were to obtain a representative and uniformly mixed sample. The soils were first mixed using standard "cone and quartering" techniques. This provided a uniform sample which was used in the characterization, Phase 1, and Phase 2 tests.

Characterization

Each characterization task had a particular objective. Characterization tasks were carried out on a portion of the homogenized sample to determine the following sample properties.

1. Bulk density
2. Air-dry moisture
3. pH determination
4. Particle size analysis
5. Gross alpha and gross beta activities
6. Plutonium 239 + 240 activity

The information obtained in the characterization stage was applied to the Phase 1 and Phase 2 test programs. Section 3.2.1 describes characterization procedures. Section 4.1.2 discusses characterization results.

3.1.2 A Primary Methods

3.1.2.1 Bulk Density Determination Objectives

The objective of bulk density determinations was to obtain a value for bulk density which could be used to relate volume and mass.

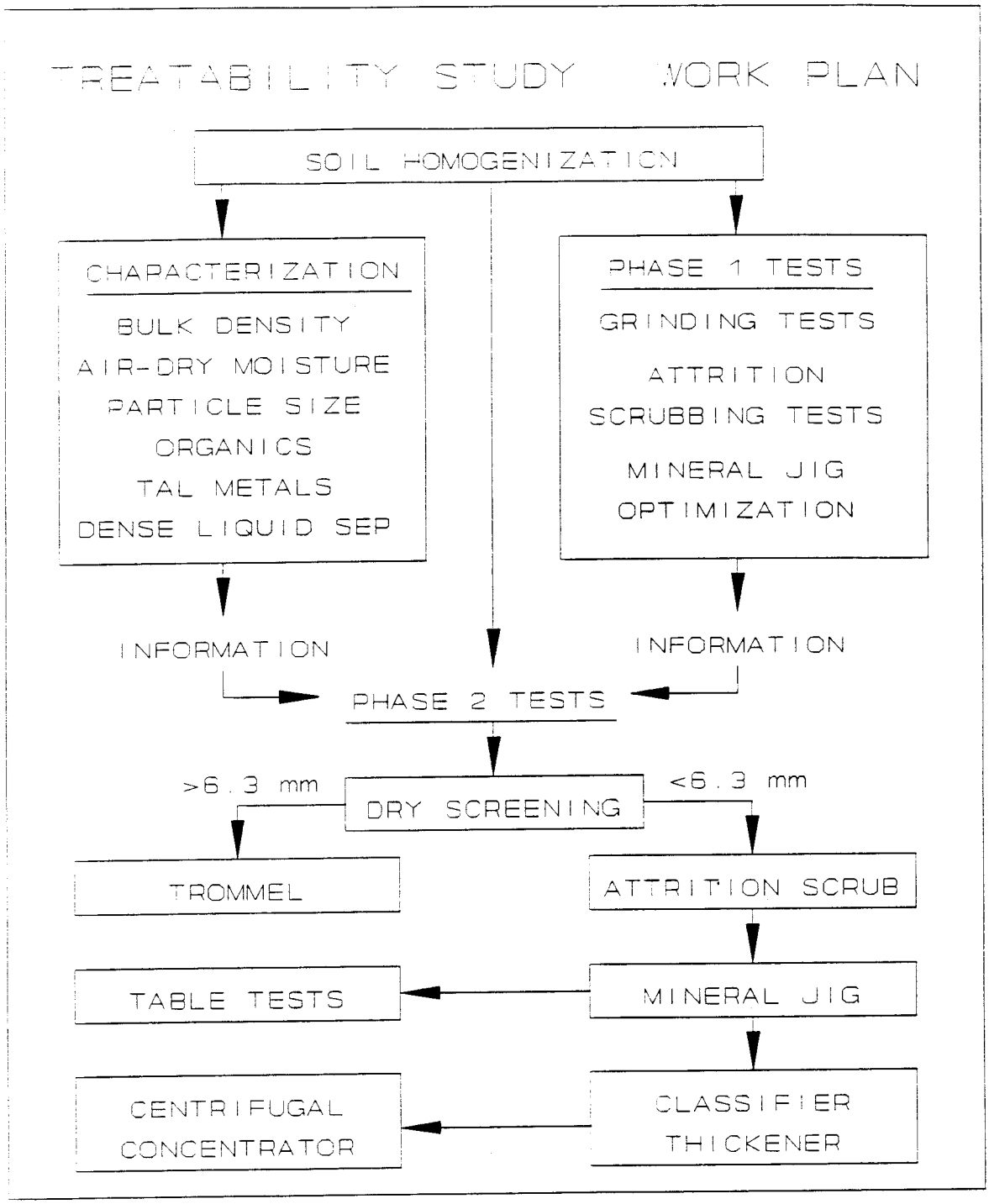


Figure 3.1 Work Plan Tasks

The bulk density of the LES&TC-CS split was obtained as specified by subsection 7.3.3.1 of the Work Plan, and in accordance with Analytical Procedure AP AWC 2, Bulk Density Determination, section 5.5.4, Volume-Filling Bulk Density Sampling.

In general, all soils have bulk densities less than the particle densities of constituent minerals (2.65 g/cm^3 Donahue; et al., 1977), and are often in the neighborhood of 1.0 to 1.5 g/cm^3. Organic material lowers the bulk density of soil.

3.1.2.2 Percent Air-Dry Moisture Objectives

The objective of measuring soil percent air-dry moisture is to obtain a value for percent air-dry moisture which gives a "working number" to use in measuring out samples from a sample lot which is in equilibrium with air of a given moisture content (relative humidity). This enables workers to remove soil samples from a container of soil and calculate the mass of solids in the subsample without oven drying the samples. As soil previously oven dried will take up moisture from the air unless sealed tightly in a desiccating environment, working with air-dry samples is a very convenient method of using subsamples.

Percent air-dry moisture was measured as specified by Section 7.3.3.2 of the Work Plan, and in accordance with Analytical Procedure AP AWC 5, Air-Dry Moisture Determination.

3.1.2.3 pH Determination Objectives

The objective for measuring soil pH was to obtain a value for the activity of protons in solutions at equilibrium with the soil.

Soil pH was measured as specified by Subsection 7.3.3.3 of the Work Plan, and in accordance with Analytical Procedure AP AWC 4, pH Determination, using both water and calcium chloride suspensions.

Suspensions made up in calcium chloride are thought to better reflect the soil pH of non-saline soils due to pH independence of soil:solution ratios and due to the flocculation of soil clays, which minimizes errors arising from liquid-junction potentials (Page, et al., 1982, Chapter 12).

3.1.2.4 Particle Size Analysis and Activity Distribution Objectives

The objective for determining the size distribution of soil particles was to provide pre-testing information on the distribution of the total mass and total activity. Additionally, the distribution of mass among size classes can be combined with data on individual soil components such as mineral types and organic matter to yield an estimate of which soil components might influence the retention and removal of plutonium from that soil. Knowledge of the distribution of americium/plutonium over different size classes can be used to identify those size classes and components which could require further remedial attention.

The distribution of soil particles and of americium/plutonium activity based upon size segregation was measured as specified by subsection 7.3.3.4 of the Work Plan, and in accordance with Analytical Procedures AP AWC 3, Particle Size Analysis, and AP AWC 19, Gamma Spectroscopic by NaI. Gamma Spectroscopy analysis by NaI is discussed in the following section.

3.1.2.5 Gamma Spectroscopy Analysis Objectives

The objective for using gamma spectroscopy was to measure ^{241}Am activity in the characterization and Phase 1 and 2 treatability tests. Rapid analysis of samples was required since many of the samples analyzed were subjected to additional test procedures after activity determination. Activity determination was performed using non-destructive gamma spectrum analysis. ^{241}Am was selected as the isotope measured. ^{241}Am is a daughter product of ^{241}Pu and can be used as an indicator of plutonium activity if the ratio of activities between plutonium and americium has been established. Results for the analyses of plutonium and americium isotopes of interest, performed to establish this ratio, are discussed in Section 4.1.2.5.

3.1.2.6 Dense Liquid Characterization Objectives

The objective for performing dense liquid characterizations was to evaluate the ability to separate dense particles of plutonium oxide from the less dense silicate soil minerals.

The separation of particles based upon their density differences was performed in accordance with TAD-LAB-I-1, Dense Liquid Characterization of Soil Samples.

This technique is often used for separating out dense precious metals from less dense silicate minerals. A liquid of controllable density, sodium polytungstate, is used to adjust the density of the medium into which soil samples are placed. Minerals more dense than the liquid, such as plutonium oxide (11.0 g/cm^3), will sink to the bottom of the container holding the liquid; materials less dense than the liquid will float on the surface of the liquid in the container.

Sodium Polytungstate is used for densities between 2.89 and 1.1 g/cm^3. This liquid is "thinned out" by adding aliquots of distilled water. The desired density of the liquid medium can be achieved either by experiment or from a (roughly) linear equation.

3.1.2.7 Pipet-Method Analysis Objectives

The objective of this method, though not required by the Work Plan, was to gather information about the silt and clay size activity distributions.

A pipet-method analysis for <45 micron diameter material was carried out in accordance with AP AWC 3, Dense Liquid Characterization of Soil Samples, subsection 5.7, Pipetting Method.

The pipet method is based upon Stoke's law of settling velocities. Variables accounted for by Stoke's law include the velocity of fall in a medium, the "equivalent" radius of a particle, the density of a particle, and the density and viscosity of the solution. Soil minerals have an average of 2.65 g/cm^3; this value is often used to determine sampling times for differing diameters. Discrete particles of plutonium oxide, with densities of 11.0 g/cm^3, are expected to fall at a faster rate than most silicates. For the purposes of this report it is assumed that most smaller plutonium oxide particles are attached to larger silicate particles and thus will have overall densities more like that of the silicates.

3.1.2 B Miscellaneous Methods

Plutonium 239 + 240 Determination Objectives

The objective of this analysis was to obtain a value for the activity level of $^{239, 240}$Pu in the starting bulk sample.

Two samples were submitted to the Lockheed Analytical Laboratory in Las Vegas to establish the ratio between $^{239, 240}$Pu and ^{241}Am.

Gross Alpha and Gross Beta Determination

The objective of gross alpha and gross beta measurements was to establish their levels for this soil.

Gross alpha and gross beta activities in soil samples were determined by independent laboratories on samples from the characterization studies and the bench scale tests. Two samples were analyzed for gross alpha and gross beta activities to establish a ratio between gross alpha and gross beta to ^{241}Am. This ratio can be used to evaluate the activity levels in the characterization and bench scale tests.

Mineralogy Analysis Objectives

The objective of mineralogical analysis was to determine the mineralogical make-up of the soil.

Quantitative evaluation of the mineralogy of the sand, silt, and clay fractions by X-ray diffraction analysis was not carried out due to the lack of laboratories which can handle plutonium containing materials. Qualitative observations are discussed in Section 4.1.2 B.

Organic Leaves and Roots Analysis Objective

The objective of analyzing the activity levels of leaves and roots was to determine the extent of activity associated with each fraction.

The unexpected high activity level for the wet sieve >37.5 mm diameter organic matter motivated the investigation of the source of this activity. A rapid, qualitative experiment was devised to assess the source of the activity (leaves or roots).

Particle Surface Photography Objectives

A photographic record of the surfaces of several pieces of gravel was made. The objective for creating this record was to gain an understanding of the potential existence of contaminated clay and silt sized particles on the surfaces of larger particles. Coatings of iron oxide, manganese oxide, carbonates, and organic matter can be found in many soils. Plutonium can be expected to be bound to the fine material as reactive surface areas are very abundant on a per gram basis compared to gravel.

Sampling Method Comparison Objectives

The objective for this comparison was to measure the activity levels of samples obtained under the two sampling methods employed in this work.

Sampling of Phase 2 testing process products was conducted, as specified by SOP AWC 101. This represented a variance from the specification in the Work Plan, Section 7.3.6.

Because the sampling performed on Phase 2 process samples varied from that stated in the Work Plan, Section 7.3.6, resampling of nine process products was conducted to compare the results of the variation.

Process Water Filter Test Objectives

The objective of this test was to gauge the filterability of activity in the settled water remaining after process run #8 by measuring the portion of activity which would pass through a filter paper.

3.1.3 Phase 1 Treatability Testing Objectives

The overall objectives of the Phase 1 tests were as follows: 1) to evaluate methods of liberating plutonium from surfaces of the soil particles by feed preparation techniques, thereby making the plutonium available for removal using gravity separation and 2) overall optimization of the gravity separation circuit. Two liberation techniques were evaluated in Phase 1 treatability testing: autogenous grinding and attrition scrubbing. Autogenous grinding was studied using a twenty centimeter lab-scale ball mill. Attrition scrubbing was evaluated using a laboratory scale attrition scrubber. Optimization of the gravity separation circuit was accomplished using magnetite and bismuth as plutonium surrogates. Section 3.2.2 discusses Phase 1 procedures. Section 4.1.3 describes Phase 1 results.

The Phase 1 treatability testing evaluated processing variables for individual pieces of process equipment to determine the optimum sequence and settings of equipment. The characterization phase indicated residual plutonium remained in all size fractions during wet sieving. Activity levels were low and enough that they did not indicate the existence of micron sized individual particles of plutonium around the size of sieve openings. Plutonium, therefore, was believed to be present as coatings, molecular size plutonium, or plutonium particles associated with clays and silts residing on the larger soil grains.

The goal of the tests was to free any attached plutonium from soil particle surfaces through feed preparation techniques and to make the plutonium available for removal using gravity separation techniques or to isolate the plutonium using size separation techniques. Two liberation techniques were evaluated in the Phase 1 treatability testing: autogenous grinding and attrition scrubbing. Each of these liberation tests are described in the following sections.

Once the plutonium bearing particles are removed from the surface of the larger soil particles, they most likely will be present on clay and silt size soil particles. These fine size particles are expected to overflow the spiral classifier and be captured in the thickener. If any larger size plutonium particles (>10 micron diameter) are present, they will be removed by gravity separation devices if they possess sufficient differences in density.

Phase 1 treatability mineral jig testing was performed for DOE at the Nevada Test Site, using density-surrogate materials of magnetite and bismuth mixed with soil (Wenstrand, et al, 1993). In these tests a known mass of magnetite or bismuth was added to the soil volume. Recovery rates were evaluated as a function of mineral jig settings. Magnetite was recovered using a high strength magnet. Magnetite was then sized and weighed to determine recovery mass. Bismuth recoveries were determined by analytical chemistry techniques. A detailed description of the test is found in the Appendix A. This test was not repeated in the interest of economics.

3.1.3.1 Autogenous Grinding Objectives

The objective of this test was to evaluate the time related function of autogenous grinding to reduce plutonium activity levels on the surfaces of >6.3 mm diameter particles in Rocky Flats soils.

3.1.3.2 Attrition Scrubber Testing Objectives

The objectives of this test were to evaluate the time related function of attrition scrubbing on removal of plutonium from the surfaces of <4.8 mm, >0.15 mm Rocky Flats soils.

This test was performed to ascertain the required residence time in the attrition scrubber for reducing levels of plutonium and freeing any particulate plutonium from mineral surfaces for recovery by the mineral jig. The activity levels remaining on soil particles following the scrubbing tests provided guidance to scrubbing times required in larger scale systems.

3.1.3.3 Mineral Jig/Spiral Classifier Testing Objectives

The objectives of these tests were to determine the optimum settings for the mineral jig and spiral classifier for recovery of liberated plutonium from soils.

The tests were performed using surrogate minerals of magnetite and bismuth to simulate plutonium particles in soil. Magnetite was selected as a surrogate due to a density lower than that of plutonium oxide. Thus,

magnetite would represent a particle which is more difficult to capture by gravity separation than plutonium oxide. Bismuth was then used to verify the test results as it has a similar density to plutonium oxide. The surrogate testing was necessary to optimize the process variables and to demonstrate recoveries since activity levels of plutonium in these Rocky Flats soils exist at relatively low levels.

3.1.4 Phase 2 Treatability Testing Objectives

The primary objective of Phase 2 was to evaluate the ability of a remediation process to reduce the activity of plutonium, gross alpha and gross beta in Rocky Flats soils using a continuous feed system. Previous investigations in 1987 indicated that the TRUclean process could remove radioactivity from Rocky Flats soils down to levels approaching the current proposed performance of 0.9 pCi $^{239+240}$ Pu/g (AWC, 1987). The treatability tests used in this study incorporate modifications to the feed preparation methods which were used in the 1987 tests.

The Phase 2 treatability study evaluated several unit operations which exploited four areas of mineral processing (feed preparation, size separation, gravity separation, and size/shape/density classification). The larger gravels were initially removed from the soil by dry screening. The material retained on the screens, after hand-picking off clumps of grass, was processed through a trommel to scrub and wash plutonium particles, clays, silts, and sands from their surfaces. The soil fraction smaller than the screen size was scrubbed in an attrition scrubber to free contaminants from the surfaces. The scrubbed slurry was then passed through a mineral jig to remove any liberated particulate plutonium from the soil. The concentrates from the mineral jig were further concentrated on a Wilfley Table to further reduce the volume of the concentrate stream. The tail from the mineral jig was processed through a spiral classifier to separate the fines and water from the sand and gravels. The spiral classifier overflow containing fine particles (<0.075 mm) was collected in a thickener. The overflow water from the thickener was recycled back to the process circuit. The fines which collected in the thickener were passed through a centrifugal gravity concentrator to remove any heavy particles. To evaluate the effectiveness of size classification, the centrifugal concentrator tails stream was treated in a hydrocyclone operated at three separate pressure settings.

Each treatability test was conducted with 90 to 110 kilograms of soil. Samples were obtained during and following the tests from process output streams to evaluate system performance. Samples obtained from the characterization and bench scale testing were sent to outside laboratories for independent analysis.

Section 3.2.3 discusses Phase 2 procedures. Section 4.1.4 describes the results of analyses conducted on samples collected during or after the Phase 2 tests to evaluate test performance.

To assist in following the logic used for the Phase 2 treatability study, a diagram of the test performed is provided in Figure 3.2. As shown in the figure, the gravels and cobbles larger than 50.8 mm were rejected in the field during the sample collection phase. The mass of this plus 50.8 mm material was weighed and determined to be 21.1% of the total soil mass. The remaining sample was sent to the Lockheed laboratories for the treatability study.

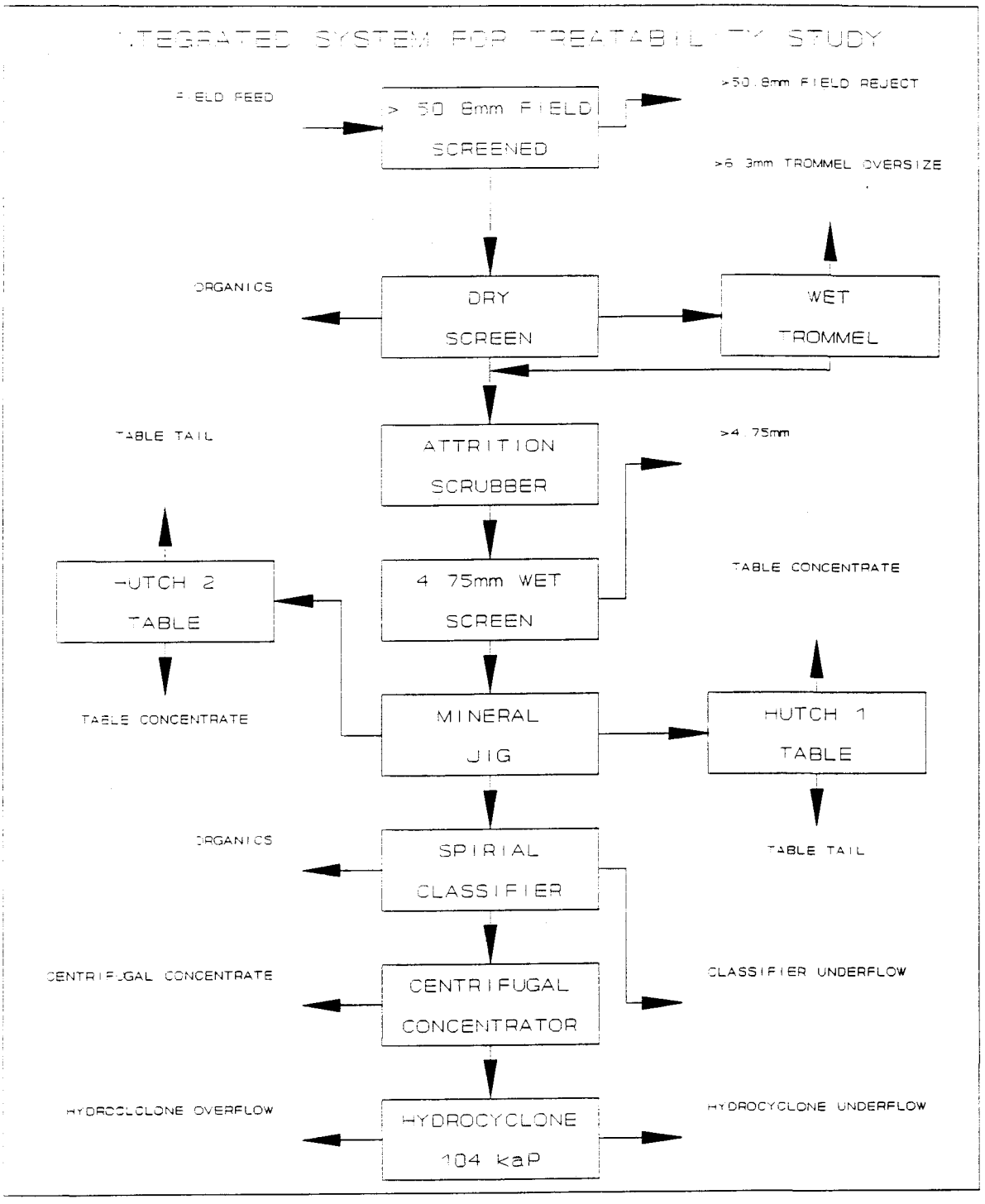


Figure 3.2 Integrated System

Following homogenization of the soil samples, the treatability tests were performed in distinct segments or groups using process separations. This allowed for combination of final results into an integrated system. The objectives for each distinct group or segment of the test is described in the following sections. The test groups or segments were:

1. Dry vibrating screening of homogenized feed (Section 3.1.4.1). Run numbers 1, 3, 5, and 7.

2. Trommel scrubbing and screening of >6.3 mm or >9.5 mm diameter dry screened material (Section 3.1.4.2). Run numbers 25, 26, 27, and 28.

3. Mineral jig separation of high density materials using attrition scrubbing to liberate plutonium particles, spiral classification to dewater mineral jig tails, and thickening to catch suspended solids (Section 3.1.4.3). Run numbers 2, 4, 6, and 8.

4. Concentration of mineral jig hutch products using a Wilfley shaker table (Section 3.1.4.4). Run numbers 9, 10, 11, 12, 13, 14, 15, and 16.

5. Centrifugal concentration and size separation of the thickener solids (Section 3.1.4.5). Run numbers 17/18, 19/20, 21/22, and 23/24.

The following explanation is presented to illustrate the relationship of the various runs performed during the tests. The >31.8 mm and >9.5 mm diameter material from run #1 were processed through the trommel as run #25. The <9.5 mm diameter material from run #1 was processed as run #2 through the mineral jig. The mineral jig concentrates from run #2 were passed across the Wilfley table as runs #9 and #10. Thickener solids from run #2 were passed through the centrifugal concentrator and hydrocyclone unit as runs #17 and #18. This run sequence was repeated for the next batch of soils. In similar fashion, run numbers 3, 26, 4, 11, 12, and 19/20 were performed in this sequence.

Information on the objects for individual tests is found in the following sections.

3.1.4.1 Dry Screening Test Objectives

The objective of this test was to remove the bulk of naturally occurring undecomposed organic matter from the soil and to provide sized feed material for the trommel tests and gravity separation tests.

The characterization portion of this project indicated that the soils contained a high level of undecomposed organic material which would interfere with the testing process and potentially elevate plutonium levels. Since the test plan provided for flexibility to optimize the feasibility study, the soil was dry screened to remove as much of these organics as possible before processing.

3.1.4.2 Trommel Test Objectives

The objective of the trommel testing was to evaluate the scrubbing and screening action of the trommel for liberating and washing contaminants

from gravels found in the Rocky Flats soils. Particles larger than 6.3 mm (1/4") were used.

3.1.4.3 Attrition Scrubbing, Mineral Jig, Spiral Classifier, and Thickener Test Objectives

The objective of this test was to liberate attached plutonium from the <6.3 mm diameter soil using attrition scrubbing and to separate any liberated plutonium from the soil using a mineral jig. Plutonium particles which are smaller than the recovery range of the mineral jig were collected in the thickener for further processing.

This test was performed to evaluate the quantity of plutonium which might be present as liberated plutonium with particle diameters above 50 microns.

3.1.4.4 Wilfley Table Test Objectives

The objective of this test was to reduce the volume of the jig concentrates and to isolate plutonium particles in as small a volume as possible.

The mineral jig hutches collect some low density sands along with the high density materials. The shaker table allows separation of these low density sands from the high density materials, thus providing a further concentration of the high density materials. The low density materials are generally considered free of liberated plutonium.

3.1.4.5 Centrifugal Concentrator and Hydrocyclone Test Objectives

The objective of the centrifugal concentrator test was to remove any liberated plutonium present in the tail product of the mineral jig which collected in the thickener. After removing any liberated particles in the range of the centrifugal concentrator effectiveness, the tail product from the concentrator was passed through a hydrocyclone. This was done to determine if a size separation can effect a concentration following removal of plutonium particles down to about 5 microns.

3.1.4.6 Post-Run Test and Analysis Objectives

Two samples from the mineral jig run #4 were subjected to further washing to determine if additional activity could be removed from the soil sample. The two samples selected for the study were sample RF2A0030 from the classifier underflow which had ^{241}Am activity of 3.32 pCi/g and sample RF2A0036 from the >4.8 mm opening diameter screen discharge which had an ^{241}Am activity of 2.31 pCi/g.

3.1.4.7 Process Water Settling Test Objectives

The objectives for performing separate tests on the water which had been used for processing was to evaluate the settling characteristics of the suspended solids.

3.2 Experimental Design and Procedures

3.2.1 Characterization Design and Procedures

Brief descriptions for the procedures used in each characterization technique are given in the following subsections. The SOP covering the particular procedure is cited.

3.2.1 A Primary Methods

3.2.1.1 Bulk Density Experimental Procedures

The procedure used for bulk density measurements was Analytical Procedure, AP AWC 2, Bulk Density Determination, Section 5.5.4, Volume-Filling Bulk Density Sampling. Briefly, a 1.67 kg grab sample was removed from the LES&T-CS soil. Sample was added to each tared beaker, taking care to avoid compressing soil. When sample overflowed a beaker, a straight edge stainless steel spatula was drawn level across the top of the beaker to remove excess soil without compressing sample down.

Beakers were then weighed and placed into the oven for drying at 105°C. After oven drying, samples were then reweighed. Calculations for bulk density and percent moisture content were recorded on the Bulk Sample Raw Data form found in Appendix A.

3.2.1.2 Percent Air-Dry Moisture Experimental Procedures

The method used for air-dry moisture determination is that of Analytical Procedure AP AWC 5. Briefly, soil was spread out on a rubber mat and exposed for 5 days to air dry. The soil was mixed each day to ensure adequate drying.

Sample was then divided, with soil being added into two tared SRCCs. The SRCCs were then weighed and placed into ovens for drying (generally two days) at 105°C until weight changes between two successive 2-hour weighings were less than 1%.

After oven drying, samples were reweighed. Calculations for air-dry moisture percentage were recorded on the Air Dry Moisture Percent form (Appendix A).

3.2.1.3 pH Determination Experimental Procedures

The procedure for measurement of soil pH is described in detail in AP AWC 4, pH Determination. Briefly, following calibration of the pH meter and electrode, 1:1 soil: solution suspensions were made up in triplicate using a 20.0 g grab sample of <2.0 mm soil, and 20.0 mL of solution. One set of triplicates was prepared using distilled water, and another set using the calcium chloride solution. Each suspension was stirred three times over a 45 minute period, which permitted the suspensions to come to equilibrium.

After suspensions settled out, the calibrated electrode was placed into the suspension, with the fritted junction setting just below the solution/air surface. The "measure" function key was activated, and following a period for the electrode to come to equilibrium with the suspension, the pH meter signaled that a stable value was reached. This value was recorded on the pH data form (attached in Appendix A).

3.2.1.4 Particle Size Analysis Experimental Procedures

The particle size and activity distribution determination is described in depth in AP TAD 3, Particle Size Analysis. Some variation from the Work Plan and Analytical Procedure, noted below, did occur. These variations involved the use of sieve nests for dry sieving and the use of pan baths for some wet sieving.

Four 9 to 10 kg fractions (36 kg total sample) were dry sieved separately in a nest of the 37.5, 25, and 19 mm diameter-opening sieves and receiving pan. Sample particles less than 19 mm in diameter were collected in a receiving pan. Sample sieve nests were shaken for 10 minutes each, after which each fraction was removed, placed into aluminum pans or SRCCs, dried, and weighed. Following the final sieve/shake, material for each sieve fraction was collected.

The material from each of the three screen fractions and the receiving pan was then split separately in the Sepor splitter. Grass material larger than 37.5 mm diameter was split separately from gravel; when several splitter chutes were covered with grass, the grass was pushed into the chutes it covered (this required some breaking of the grass into sections). Final split samples were subsampled and analyzed by gamma spectroscopy for ^{241}Am to yield an estimate of beginning activity balances.

Following weighing and gamma analysis, samples from the three sieve fractions were returned to their respective sieves and sieved with water, which served to remove smaller particles adhered to larger particles or contained in aggregates. Wet sieving was carried out until the rinse water became clean. Dry sieved subsamples which were analyzed by gamma spectroscopy were wet sieved separately from the bulk fraction, and then reanalyzed by gamma spectroscopy for comparison. These samples were returned to their respective sieve fractions. Each fraction was then split using the sample splitter. The final split sample was analyzed for ^{241}Am activity. All 37.5, 25, and 19 mm diameter samples were archived.

The less-than 19 mm diameter material previously wet sieved was combined with the less-than 19 mm diameter dry sieve material and mixed. The composited material was then split down to a 4 kg sample. The 4 kg sample was then dry sieved through a nest of 9.5 mm to 850 micron diameter-opening sieves and the soil fraction smaller than 850 micron diameter collected in a receiving pan. The minus 850 micron diameter soils were dry sieved through a sieve nest comprised of 425 and 300 micron diameter-opening sieves, with the soil fraction smaller

than 300 micron diameter collected in a receiving pan. Separate nests were necessary due to the capacity limits of the sieve shaker. All material in all fractions was analyzed for mass and activity following sieving.

After activity analysis, samples were returned to their respective sieve fractions for wet sieving. The 9.5, 6.3, and 4.0 micron diameter fractions were wet sieved with a fine spray of water. The remaining fractions were, however, wet sieved in a "pan bath" which consisted of an aluminum pan, large enough to accommodate the sieve, half-filled with water. This method has been found to be very time effective for fractions from 2.0 mm to 150 micron diameter, and involves a swirling motion using a plastic spatula. Particles are drawn very rapidly over sieve openings. The fall of particles in the liquid medium is very rapid, and is described by Stoke's law of settling velocities (Donahue, et al., 1977).

Wet sieved fractions were dried, weighed, and analyzed by gamma spectroscopy. All sample fractions with particle diameters 300 micron or larger were archived. Material less than 300 micron diameter (a 2.3 kg sample) was then dry sieved in a sieve nest on the shaker, analyzed for activity, then wet sieved, dried, and reanalyzed. All samples, other than two <45 micron diameter samples removed for pipet method analysis and dense liquid characterization, were archived.

3.2.1.5 Gamma Spectroscopy Analysis Experimental Procedures

Analysis by gamma spectroscopy is described more thoroughly in AP AWC 19, Gamma Spectroscopic Analysis by NaI. Prior to analysis, samples were dried in an oven at 105°C to remove free and adsorbed moisture. All samples were placed into tared SRCCs. Each sample was weighed and assigned a sample identification number.

Each sample was counted in a region of interest (ROI) centered around the 60 keV peak for ^{241}Am. Total peak counts for the ROI were integrated and converted to americium counts by determining counting efficiency using a NIST traceable standard. The length of counting times was selected to provide statistically significant counts.

Calibration efficiencies for the gamma spectrometer were obtained daily using a NIST traceable ^{241}Am standard. The efficiency obtained by the standard was then applied to the unknown samples counted on that day.

3.2.1.6 Dense Liquid Characterization Experimental Procedures

The procedure used varied slightly from TAD-LAB-I-1, as centrifugation was used instead of gravity settling. The sample used was a "grab" sample from the <45 micron diameter material and may not be representative of the overall fraction.

Two-fifty gram replicate grab samples were removed from a less-than 45 micron diameter wet sieved sample and placed into the 250 mL centrifuge bottles. A 50/50 mL polytungstate/mL distilled water solution

was made up for each replicate. One hundred milliliters were added directly into the sample. The density measured 1.9 g/cm^3 for each replicate solution.

Samples were shaken for 1 hour then centrifuged at 3100 rpm for 5 minutes. Samples were then removed and allowed to stand for several minutes. The layer formed at the top was carefully removed using the stainless steel spatula. As suspended material remained in the samples, standing liquid was decanted off from the settled material and added to the material from the top layer.

Material removed was mixed with water at 3 times the volume of removed sample, then filtered on a Buchner filter flask apparatus. Copious amounts of water were added during filtering to remove residual polytungstate. Filtered solids were placed into a tared SRCC and dried in an oven at 105°C. Liquid which passed through the filter was also placed into a separate SRCC and dried.

When dried, samples were reweighed and then analyzed by gamma spectroscopy.

Pure liquid polytungstate (2.9 g/cm^3 was added back into the sink product in the centrifuge bottles to replace the mass of liquid and solids removed. The new density was calculated as approximately 2.6 g/cm^3. Samples were reshaken, centrifuged, and filtered.

This sequence was repeated once more. The final density of the last run was about 2.9 g/cm^3. Sink product from the last treatment was washed and filtered at the end of this sequence. All samples and filtered liquid products were analyzed by gamma spectroscopy.

3.2.1.7 Pipet-Method Analysis Experimental Procedure

One hundred grams of a less-than 45 micron diameter sample chosen for its average activity level was added to 400 mL of distilled water in a dispersion cup. The mixture was placed into a dispersion cup and onto a Hamilton Beach blender at medium speed for 5 minutes. Following mixing, the sample was placed into the sedimentation cylinder, 10 mL of stock hexametaphosphate solution was added, and the mixture was brought up to the 1 L level.

After standing for 2 hours to come to room temperature, the cylinder was covered with plastic, inverted, and shaken by turning over rapidly for 2 minutes. At the end of mixing, the timing for sample removal began. Samples were removed at the appropriate time intervals for 20, 5, and 2 micron diameter fractions. Samples were placed into SRCCs, oven dried at 105°C, weighed, and analyzed by gamma spectroscopy. Room temperature remained at 23°C throughout the analysis.

3.2.1 B Miscellaneous Methods

Organic Leaves and Roots Analysis Experimental Procedures

The leaves of the grass found in the greater than 37.5 mm fraction were broken off above the stem and root section and placed into a separate pan. This process generated some loose dust, both falling off of the roots and apparently coming off of "shattered" sections of the dried grass in the containers.

The dust from both the roots container and the leaves container was combined into one SRCC. Leaves were placed into another SRCC, and roots into a third SRCC. Each sample was dried at 105°C, weighed, and analyzed by gamma spectroscopy. Two samples of the grass material was processed in this manner.

Sampling Method Comparison Experimental Procedures

Sampling methodologies are described in Section 3.4. Briefly, the SOP AWC 101 method requires a thorough homogenization (by unspecified means) and the removal of a sample. Two samples were removed from most samples as duplicates. In contrast, Work Plan Subsection 7.3.6 requires the use of a splitter for all slurry and dry samples. A comparison of the two procedures was carried out on Phase 2 generated samples.

Process Water Filter Test Experimental Procedures

To gauge the filterable activity in the process water, an approximately 3 L sample of process water was taken 5 days after Run #8. This water was vacuum filtered through a Whatman #42 (nominal pore opening of 2.5 micron) filter paper in a Buchner Funnel. Both the filter paper and filtered water were placed in separate SRCC's and dried down in an oven at 105°C.

3.2.2 Phase 1 Design and Procedures

Brief descriptions for the procedures used in Phase 1 testing are given in the following subsections. The SOP covering the particular procedure is cited.

3.2.2.1 Autogenous Grinding Experimental Procedures

The procedure for conducting autogenous grinding tests is described in SOP TAD 603, "Tumbling Tests."

Briefly, autogenous grinding tests were performed on the >6.3 mm diameter soil fractions using a lab-scale ball mill to determine optimum grinding times. Feed for the test was wet sieved through a 6.3 mm opening size screen. Pulp density of the slurry was controlled by water addition at the beginning of each test. The >6.3 mm diameter fraction was placed in the mill and tumbled below critical speed for 1 minute. The material was removed from the mill and wet screened at 6.3 mm diameter to separate off liberated material and to determine activity levels on the washed fraction. The 6.3 mm diameter material was returned to the mill and grinding continued for an additional minute. Following grinding, the material was again removed, wet sieved, and activity

determined. This procedure was repeated for 5, 10, and 20 minutes to develop the graph of activity concentration as a function of grinding time.

3.2.2.2 Attrition Scrubber Testing Experimental Procedures

The procedure for conducting attrition scrubbing tests is described in SOP AWC 210, "Operation of the Laboratory Attrition Scrubber."

Briefly, attrition scrubbing tests were performed using a laboratory scale attrition scrubber. For these tests the feed material was prepared by isolating a soil sample comprised of <4.8 mm and >0.15 mm sized soil grains. Initial activity measurements were made. The sample was then placed in the scrubbing chamber along with water to obtain a slurry density of 65% solids by weight. The rotational speed was maintained at 900 RPM for the tests. The sample was then scrubbed for 1 minute and the sample removed. The sample was then wet sieved at >0.85 mm, <0.85 mm, >0.30 mm, and <0.30 mm >0.15 mm. Each size fraction was measured for activity level before recombining for the additional attrition scrubbing time.

3.2.2.3 Mineral Jig/Spiral Classifier Experimental Procedures

The procedures for these tests are given in SOP AWC 202, "Operation of the Gravimetric Separator (Mineral Jig)"; SOP AWC 211, "Operation of the Spiral Classifier"; SOP AWC 207, "Operation of Trommel"; and SOP AWC 213, "Operation of Thickener."

Briefly, these tests were performed by adding known quantities of magnetite or bismuth to feed soils. The soils were then processed through the trommel, mineral jig, spiral classifier, and thickener to evaluate recoveries as a function of equipment settings. Following these tests, the optimum settings were obtained for recovery of plutonium oxide from soils. Surrogate particle sizes ranged from 20 microns to 300 microns for the tests.

3.2.3 Phase 2 Design and Procedures

Brief descriptions for the procedures used in operating each individual piece of processing equipment are given in the following subsections.

The SOP covering the particular procedure described is listed for all equipment except the dry screen.

3.2.3.1 Dry Screening Test Procedures

The dry screening tests (designated as run numbers 1, 3, 5, and 7) involved passing the soils over a double deck vibrating screen and collecting the three resulting streams for further processing. For run #1 the screen opening sizes were 31.8 mm and 9.5 mm. For run numbers 3, 5, and 7 the lower screen opening size was reduced to 6.3 mm. The 31.8 mm opening screen successfully collected a large portion of the undecomposed organic material along with the <51 mm, >31.8 mm

gravel. The organic material collected into large clumps and was separated from the gravel. The dry screen used in the test was totally enclosed and dust controlled at the inlet using a snorkel ventilation system which was filtered by a HEPA ventilation system.

Pursuant to the test plan, at least 90.8 kg of soil was used in each of the three required tests. Prior to the test run, the soil for each test was weighed, providing necessary measurements for determining a mass balance following the test. Moisture content was also measured. For these tests, duplicate samples were obtained of the undecomposed organics, >31.8 mm and >9.5 mm or 6.3 mm streams. This allowed evaluation of variations in activity which could be expected in sampling and soil surface conditions.

3.2.3.2 Trommel Test Experimental Procedures

The procedure for these tests is given in SOP AWC 207, "Operation of the Trommel."

The soil was introduced into the scrubbing section of the trommel using a vibratory feeder. The angle of tilt on the trommel is variable to allow different scrubbing times in the scrub section of the trommel. The trommel was positioned at a 0° angle to maximize scrubbing times. As the soil progressed through the trommel it reached the screening section where a separation was made between the >6.3 and <6.3 mm diameter material.

The trommel tests were performed as test runs 25 through 28. The feed for the tests utilized the two combined coarse fractions from the dry screening tests (>31.8 and <31.8 mm >9.5 mm; or >31.8 and <31.8 mm, >31.8 mm).

3.2.3.3 Attrition Scrubbing, Mineral Jig, Spiral Classifier, and Thickener Test Experimental Procedures

The procedures for operation of the equipment described in this section are given in SOP AWC 209, "Operation of the Attrition Scrubber"; SOP AWC 202, "Operation of the Gravimetric Separator"; SOP AWC 211, "Operation of the Spiral Classifier"; and SOP AWC 213, "Operation of Trommel."

Feed material was introduced into the system using a vibratory feeder. The feed material was loaded into the feeder and a calibration obtained as a function of vibratory feeder settings to obtain the desired feed rate for the tests. The vibratory feeder metered the soil directly into the attrition scrubber where recycle water was added to obtain a pulp density of about 65% by weight. Water addition rates for the scrubber were calculated based on system feed rate.

After attrition scrubbing, the soil slurry was passed over a vibrating screen which contained a 4.8 mm opening diameter screen. Soil material collecting on the 4.8 mm opening diameter screen was washed with

recycle water on runs 2, 4, and 6. Test run #8 used tap water to wash gravels and organics on the screen. Upon completion of the test run, the >4.8 mm opening diameter fraction was weighed and sampled. The moisture content of the sample was also determined to calculate a dry mass of the stream. Soil passing the 4.8 mm opening diameter screen was fed directly to the mineral jig.

The settings for the mineral jig were optimized using surrogates in soil. This procedure provided a means by which higher concentrations of high density materials could be used to precisely determine the settings for optimum recoveries. Based on these tests, the mineral jig and classifier settings were determined for the treatability test. The hutch flow rate was set at 11.3 liters per minute on each hutch cell. The mineral jig stroke length was set at 0.95 cm with a frequency of 150 cycles per minute. Mineral jig ragging used 0.47 cm^3 steel balls at a depth of 1.9 cm. The above variables were physically measured at the beginning of the test or were monitored with flow meters to maintain critical flows.

Mineral jig concentrates were continuously removed from the hutches and collected in containers which were weighed and sampled following completion of the test. Since the hutch concentrates were saturated with water, the moisture content of the hutch product was also measured to allow determination of total dry mass.

Following removal of liberated high density materials by the mineral jig, the mineral jig tails flowed to a spiral classifier used to dewater the >0.075 mm soil fraction. The dewatered classifier underflow was collected in a container during the test run. Upon completion, the sample was weighed and sampled for moisture content and activity. The overflow from the spiral classifier was passed through a 0.15 mm diameter opening screen to remove organics which floated in the classifier pool. The overflow from the spiral classifier was collected in the thickener. When the test run was completed the spiral classifier was cleaned to account for all soil mass in the test.

The thickener was used to remove soil particles from the water, thus permitting the recycling of water during the tests. When each test run was completed, all suspended solids were allowed to settle overnight. Water was decanted the following morning. All water was eventually routed through the thickener for concentration and removal of fine soil particles. These thickened solids were later removed and sampled for moisture content and activity levels.

Hutch materials collected in this test were further concentrated in shaker (Wilfley) table tests. The thickener solids were subjected to further testing in a centrifugal concentrator and size separated in a hydrocyclone, as discussed in Section 3.2.3.5.

3.2.3.4 Wilfley Table Test Experimental Procedures

The procedure for operation of the Wilfley shaker table is given in SOP AWC 204, "Operation of the Shaker Table."

The mineral jig hutch concentrates were further reduced in volume by passing the concentrates over the Wilfley Table. The hutch slurries and wash water were fed to the upper edge of the Wilfley side-sloping table. As the suspension of materials moved across the table it was caught and formed pools behind the longitudinal riffles. The differential shaking action caused reverse size classification and specific gravity stratification. The outcome was that pools of slurry became arranged so that similar specific gravity particles arrange vertically according to size. Once the bed formed, the addition of more slurry and the action of the flow of cross water enabled shearing of the top layers of the stratified slurry, thereby forcing the lower specific gravity and coarser particles to roll over the riffles toward the downslope side of the table. The height and depth of the riffles and the beds decreased from the shaker mechanism end to the heavies discharge end of the table. This feature allowed continuous selective shearing of the increasingly finer sized and higher density particles as these particles moved longitudinally along the table. The final phase of concentration was achieved on the unriffled section of the table at the concentrated discharge end. Here, the slurry, consisting of only the lower strata of the beds formed behind the riffles, was carefully washed by a smooth film of cross water which moved the larger particles of a given specific gravity down the slope faster than finer particles of the same specific gravity. The mineral jig concentrates were then fed to the shaker table.

Wash water, feed rates, and critical operating parameters were recorded on the run sheets provided in the procedure. Table concentrates collected during the run were weighed and sampled for moisture content. The tail products were also collected in a container, weighed, and sampled to obtain mass and activity balances.

3.2.3.5 Centrifugal Concentrator and Hydrocyclone Experimental Procedures

Procedures for the operation of the centrifugal concentrator and the hydrocyclone can be found in SOP AWC 201, "Operation of the Falcon Concentrator Model B6," and SOP AWC 212, "Operation of the Hydrocyclone Test Unit."

Representative samples from the thickener were subjected to further gravity separation using a centrifugal concentrator. Centrifugal concentrators take advantage of the difference in specific gravity between plutonium particles and tail particles to effect a separation. A slurry stream is directed into a cone rotating at sufficient RPM to impact in excess of 300 G's to the material being processed. The centrifugal force magnifies the difference in specific gravity and the cone geometry facilitates retention of plutonium particles, while lower specific gravity particles are rejected to the concentrator tail.

Following the test run the centrifugal concentrator was stopped and the concentrate removed from the cone. The tail product from the centrifugal concentrator was transferred to a hydrocyclone test unit to evaluate a size separation which could further concentrate any of the plutonium particles. The hydrocyclone test unit used a 5.0 cm hydrocyclone and

was operated at inlet pressures of 104, 207, and 414 kPa during the tests.

Samples for the centrifugal concentrator tests were obtained by removing equal volumes of suspended soil slurries from each thickener underflow container. The sample was transferred to a container and water added to obtain the specified pulp density for the test. The contents of the container were suspended with a mechanical stirrer while being pumped to the centrifugal concentrator. The centrifugal concentrator tail was collected in a container and passed through the concentrator three more times to simulate conditions in a centrifugal concentrator with continuous concentrate removal. Following the last cycle, the centrifugal concentrator tail was transferred to the hydrocyclone test unit for further tests. Upon completion of the tests, the concentrate collected in the centrifugal concentrator was removed, weighed, and sampled for moisture content and activity measurements.

The hydrocyclone unit is designed to continuously recycle the contents of the test sample. Cyclone overflow and underflow samples are simultaneously removed to evaluate performance.

3.2.3.6 Post-Run Tests and Analysis Experimental Procedures

The two samples withdrawn from Run #4 were subjected to further washing to determine if additional activity could be removed from the sample. One sample was removed from the classifier underflow stream and had an initial activity of 19.1 $pCi^{239, 240}Pu/g$. The second sample was taken from the +4 mesh vibrating Sweco screen and had an initial activity of 13.3 $pCi^{239, 240}Pu/g$.

The >4.8 mm opening diameter sample was placed in a 4.8 mm opening diameter sieve and thoroughly washed with water. Organics were hand picked from the sieve and combined with the <4.8 mm opening diameter fraction. The classifier underflow sample was wet sieved in a 150 micron diameter-opening sieve. Organics were also hand picked from this sample.

3.2.3.7 Process Water Settling Test Experimental Procedures

Following each run, the process water was allowed to settle overnight and the clear water fraction decanted for reuse in the next test run. The majority of the process water was used in the mineral jig tests and typically involved a 760 L circulating water volume. Samples of the recycle water were obtained at the end of tests to evaluate the settling characteristics of the suspended solids. Solids were resuspended by stirring and agitation. Upon removal of the stirrer, the timer was started. At various intervals, the depth to liquid/pulp interface and to clear/cloudy interface was measured.

3.3 Equipment and Materials

3.3.1 Characterization Equipment and Materials

Equipment and materials used for a particular set of experiments is described in the following paragraphs. Except where noted, distilled water was used in all experiments.

3.3.1 A Primary Methods

3.3.1.1 Bulk Density Determination Equipment & Materials

Two graduated plastic beakers cut level were used to measure sample volume; the measured volumes of the modified beakers were 140 cm^3 each. A straight edge stainless steel spatula was used to level soil to the rim of the modified beakers. Samples were weighed on a Mettler PM 400 top-loading balance and dried at 105 ± 5°C in a Fisher Scientific Isotemp Oven, Model 655G.

3.3.1.2 Percent Air-Dry Moisture Determination Equipment & Materials

Two Standard Radiological Counting Containers (SRCC) were used as containers. Samples were weighed on a Mettler PM 400 top-loading balance and dried at 105° ± 5°C in the Fisher oven.

3.3.1.3 pH Determination Equipment & Materials

Determination of soil pH was conducted using an Orion model 420A pH meter, measuring pH to ±0.01, and an Orion Sure-flow combination pH electrode, model 8165BN. Standardization was performed prior to each measurement or each continuous series of measurements using VWR brand buffer solutions of pH 4.0, 7.0, and 10.0. All standards and samples were placed in glass beakers, with glass stirring rods used for mixing. Bottled distilled water and 0.01 M $CaCl_2$ were used to make up the soil suspensions.

3.3.1.4 Particle Size Analysis Equipment & Materials

Samples were sieved in 20 cm diameter, 5 cm height brass Tyler sieves. Dry sieving used a Portable Sieve Shaker, Model RX-24; adjoining sieves were taped together with 5 cm duct tape to minimize release of airborne material. Sieve sizes used are specified in Subsection 7.3.3.4 of the Work Plan. A non-convective drying oven was used at 105°C. Temperature was verified using an Ertco glass thermometer. Aluminum Standard Radiological Counting Containers (SRCC), 300 cm^3 volume, were used to store and dry sieve fractions, and to hold samples during gamma spectroscopic analysis.

Distilled water and plastic spatulas were used during wet sieving to pass soil through the sieves. Round, 20 cm aluminum pans were used to collect the soil fraction which passed through the bottom sieve. For pipet analysis of the <45 micron diameter fraction, the soil was dispersed

using sodium hexametaphosphate and a Hamilton Beach model H-4260 dispersion blender with an 800 mL dispersion cup. This dispersed solution was then placed in a 1 L sedimentation cylinder where samples were removed at the required settling time using 25 mL Mohr pipets.

Samples were split using either a Sepor splitter with openings of 63.5 mm or a Miners Inc. model H-3987 splitter with openings of 25.4 mm.

3.3.1.5 Gamma Spectroscopy Analysis Equipment & Materials

All samples analyzed for gamma radiation were placed into SRCCs and sealed. Each sample was counted using an Ortec Gamma Scintillation Spectrometer. Detection was by means of a 7.6 cm by 7.6 cm NaI crystal detector. Signal processing and amplification was performed with an Ortec 92X Spectrum Master amplifier and multichannel buffer (MCB) hardware. Data display and conversion was accomplished using Ortec MCA emulation and gamma-ray spectrum analysis software.

3.3.1.6 Dense Liquid Characterization Equipment & Supplies

Sodium polytungstate (density = 2.89 g/cm^3) was obtained from Geoliquids, Inc. Polyethylene, 250 mL, wide mouth centrifuge bottles were used instead of glass beakers (which may break), as centrifugation more readily effects the separation of very fine (<45 micron diameter) materials. Samples were weighed out on a Mettler PM400 top loader balance; polytungstate/water mixtures were mixed in a 250 mL volumetric flask. Samples were centrifuged in a Fisher Scientific Marathon 21K centrifuge. Other equipment included a stainless steel spatula, a reciprocating shaker, a Buchner filter funnel, Whatman 42 filter paper, 1 L side arm flasks fitted with pre-cut stoppers, and a vacuum pump.

3.3.1.7 Pipet-Method Analysis Equipment & Materials

Equipment used for pipet-method analysis includes a 1-L sedimentation cylinder, a 25 mL Mohr pipet, SRCC's, a Hamilton Beach blender and dispersion cups, a pipet bulb, a clamp and ring stand, a drying oven, and a balance. Stock solution of 50 g/L sodium hexametaphosphate was used as a dispersing agent.

3.3.1 B Miscellaneous Methods

Particle Surface Photography Equipment and Materials

Photography was conducted with an Olympus SZ-PT photo-optical microscope using reflected light provided both normal to the material and as adjustable for side lighting using light pipes. Exposure times were conducted manually. Polaroid 4 x 5 black and white film was used.

3.3.2 Phase 1 Equipment and Materials

3.3.2.1 Autogenous Grinding Equipment and Materials

The equipment used for this test consisted of a 20 cm diameter sealed drum (Lortone Model 12NR) which was rotated on a set of rollers (Lortone Model QT12) at a rate below the critical speed. Critical speed is the drum rotation which would impinge the soil to the outer surfaces of the drum by centrifugal force.

3.3.2.2 Attrition Scrubber Testing Equipment and Materials

The equipment used for these tests consisted of a laboratory Denver Flotation Machine (Model 533000, Serial Number [SN] 100065586) fitted with an attrition scrubber attachment.

3.3.2.3 Mineral Jig/Spiral Classifier Equipment and Materials

Equipment for the tests included a trommel, mineral jig, spiral classifier, and thickener (described in Section 3.3.3.3). The primary variables evaluated in the test included flow rate to the mineral jig hutches, stroke length, and stroke frequency. A detailed flow sheet of equipment used in the test is found in Figure 3.3.

3.3.3 Phase 2 Equipment and Materials

3.3.3.1 Dry Screening Test Equipment and Materials

The equipment used in the dry screening test consisted of a Smico vibrating screen (Model 91-939, SN 21772). The Smico screen has a fully sealed, dust tight enclosure for containment of soil. The unit has provisions to install two 22.9 x 91.4 cm screens, allowing separation of soils at two screening levels. The screen is operated in a counter flow rotation which provides the best size separation. A photograph of the Smico screen is shown in Appendix A.

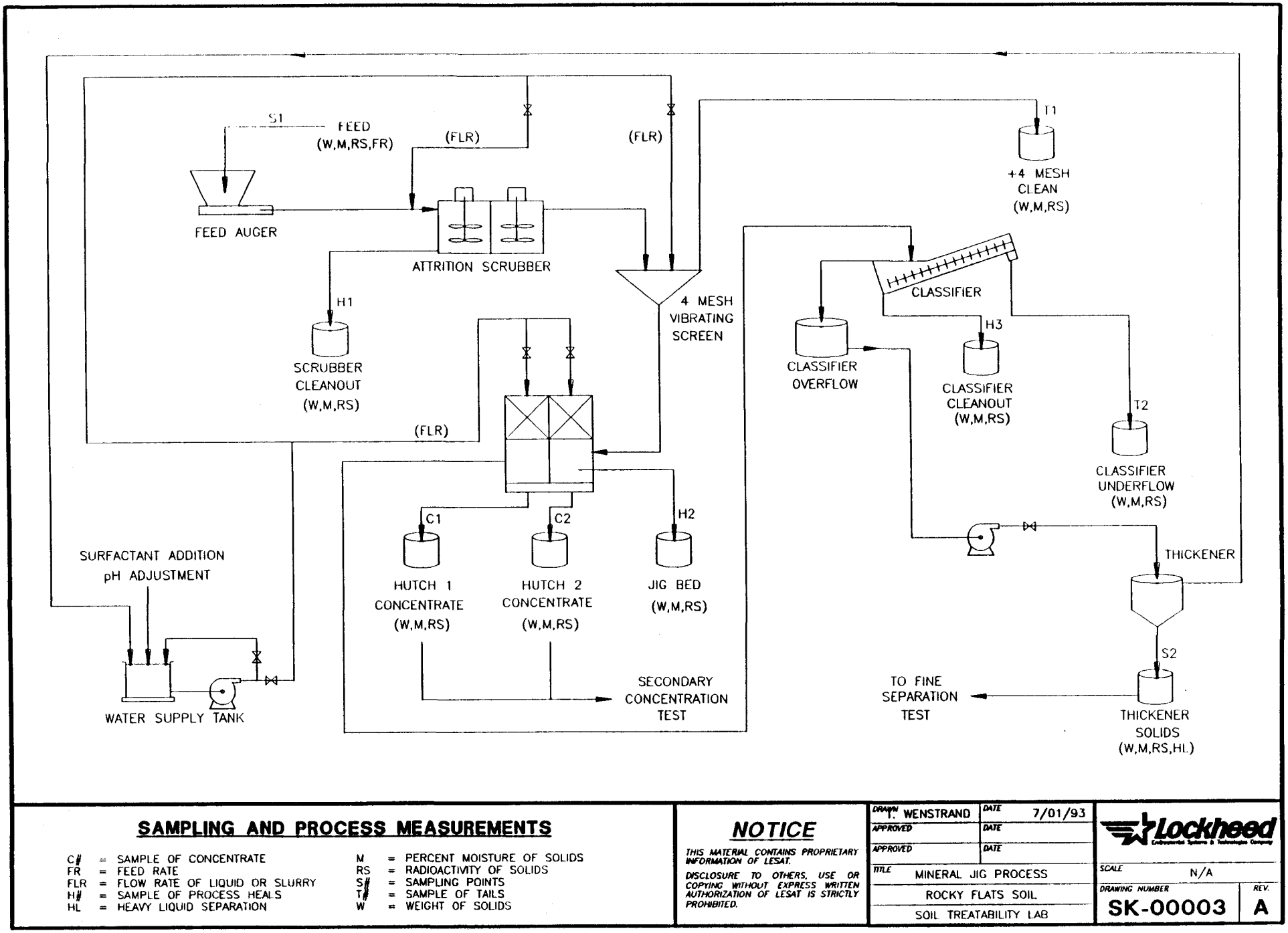
3.3.3.2 Trommel Test Equipment and Materials

The trommel is a screening/scrubbing device which is used to scrub contaminants from soil and divide the soil into two size fractions. The trommel was custom built by Goldfield, Inc., using a Boston Gear motor. The trommel contains a rotating drum which is divided into two sections consisting of an enclosed scrubbing drum and a cylindrical screen. For the treatability tests the trommel contained a 6.3 mm size opening screen. A spray bar is located inside the entire length of the trommel drum to wash the soil as it traverses the length of the drum.

3.3.3.3 Attrition Scrubbing, Mineral Jig, Spiral Classifier, and Thickener Test Equipment and Materials

For the mineral jig tests, an equipment train was assembled which consisted of a Goldfield vibratory feeder (using a Syntron magnetic feed

Figure 3.3 TRUclean Process Configuration



SAMPLING AND PROCESS MEASUREMENTS

C# = SAMPLE OF CONCENTRATE
FR = FEED RATE
FLR = FLOW RATE OF LIQUID OR SLURRY
H# = SAMPLE OF PROCESS HEALS
HL = HEAVY LIQUID SEPARATION
M = PERCENT MOISTURE OF SOLIDS
RS = RADIOACTIVITY OF SOLIDS
S# = SAMPLING POINTS
T# = SAMPLE OF TAILS
W = WEIGHT OF SOLIDS

DRAWN T. WENSTRAND	DATE 7/01/93	Lockheed
APPROVED	DATE	
APPROVED	DATE	
TITLE MINERAL JIG PROCESS		SCALE N/A
ROCKY FLATS SOIL		DRAWING NUMBER SK-00003 REV. A
SOIL TREATABILITY LAB		

vibrator), attrition scrubber, Sweco vibratory screen (Model LS18533, SN 25592-C-1292), mineral jig, spiral classifier and thickener. An illustration of the test equipment is shown in Figure 3.3.

The attrition scrubber was manufactured by Hazen Quinn (SN HQ168-1854). The attrition scrubber is designed to remove surface films and coatings from individual soil grains and break up clay agglomerates or soil cementations. This was accomplished by subjecting the soil particles to shear forces generated by opposing pitch impellers in the scrubber. The scrubber contained two cells, which required the soil slurry to pass below a baffle separating the two cells reducing the possibility of short circuiting. Scrubbing time was thereby maximized in the cells. The resulting currents induced by the impellers cause the soil grains to collide against each other, breaking up softer materials and removing coatings from the harder larger sand grains.

The mineral jig was custom built by Goldfield, Inc. The mineral jig is designed to cause a separation of materials with different specific gravities. This is accomplished by passing a slurry over a screen and subjecting the screen beds to a vertical hydraulic pulsation. This pulsation momentarily expands the beds and allows the heavier materials to work toward the bottom of the bed and lighter materials to the top of the bed. Heavier materials, or concentrate, that are finer than the screen opening will gradually pass down through the beds and the screen into the hutch, or lower compartment, from which it is continuously discharged. The lighter material, or tailings, will flow over the top of the beds and be rejected over the end of the mineral jig. The mineral jig has many variables which effect the recovery obtained in the device.

A Hazen Quinn spiral classifier (SN HQ168-1819) was used to separate the gravels and sands from the water and fine particles. The spiral classifier is a device which separates soil particles according to size and specific gravity. The spiral classifier consists of an inclined tank in which a spiral, mounted parallel to the tank bottom, rotates without contacting the sides or bottom of the tank. The spiral structure provides the necessary pool agitation and conveys the settling solids to the discharge. The slurry is introduced at the pool through the side wall. The pool level is maintained by adjusting the height of the overflow weir. The pool area permits particles larger than the separation size to settle depending on the density, shape of the particle, turbulence, density and viscosity of the slurry within the pool. The smaller particles travel with the classifier overflow over the weir. The larger particles sink to the bottom of the pool and are elevated up the incline by the spiral auger to the discharge chute.

The thickener (Hazen Quinn SN HQ168-1731) is a device used to settle suspended solids to the bottom of a tank and remove settled solids through a bottom discharge in the tank. The settled solids are maintained in a fluid state at the bottom of the tank by a slow moving agitation bar. Particle settling in the thickener is a function of particle size, density, particle shape, and density and viscosity properties of the water.

3.3.3.4 Wilfley Table Test Equipment and Materials

The Wilfley table (Model 13A, SN 91720) was set up by experimentally determining measured feed and wash water flow, measured longitudinal stroke length and frequency, and measured longitudinal slope and table incline. Jig concentrate is fed into the table feed trough at the point where feed water carries the feed onto the top corner of the table deck.

The Wilfley table consists of a side-sloping table upon which is mounted longitudinal riffles for modifying the travel of particles across the table deck. The table is mechanically shaken to cause differential movements of particles of varying sizes and densities.

3.3.3.5 Centrifugal Concentrator and Hydrocyclone Test Equipment and Materials

The centrifugal concentrator is a gravity separation device designed to separate heavy particles from lighter particles by use of centrifugal force. A Falcon Model B6 (SN B06A-901) was used for this work. The equipment consists of a revolving bowl which is rotated up to a force of 300 Gs. The geometry of the inner side of the bowl is shaped to catch heavy materials. During operation, lighter material works up the inside of the bowl surface and exits the concentrator. Following operation, the bowl is removed to access heavy materials collected along the sides of the bowl.

A feed tank, receiver tank, various piping and fittings, a stopwatch, decantation cylinder, and scale were also used.

The hydrocyclone used was a Mozley, C700 Hydrocyclone Test Rig using a 5 cm diameter hydrocyclone for d_{50} cut points down to 5 to 10 micron diameters. The hydrocyclone inlet pressure was operated from 104 to 414 kPa pressures. Various piping, fitting, feed, and receiver tanks were used.

3.3.3.6 Post-Run Tests and Analysis Equipment and Materials

Post-run testing and analysis was conducted using 4.8 mm and 0.15 mm opening diameter brass sieves. Distilled water (or fresh tap water where noted) was used for washing. All samples were weighed on balances described previously.

3.3.3.7 Process Water Settling Test Equipment and Materials

Process water was scooped from the recycle tanks using SRCCs and deposited into 4L glass beakers. Samples were resuspended using a plastic spatula for stirring and agitation. A laboratory timer recording to tenths of a second was used to measure setting times.

3.3.3.8 Support Equipment and Materials

Several pieces of support equipment were utilized in the treatability test: hoppers to contain feed, containers for water retention, pumps to supply

soil slurries to equipment, agitators to maintain soils in suspension, filters to remove particulates from water, and scales to weigh samples. Scales were calibrated with weights to verify accuracy of measurements. Flows from pumps were monitored with flow meters and values recorded on appropriate data sheets. Vibrating feeders were calibrated using actual Rocky Flats soils used in the tests.

3.4 Sampling and Analysis

Sampling and analysis was conducted during this project on the initial soil as received, during the characterization phase, during Phase 1 testing, and on feed and process streams throughout Phase 2 testing.

3.4.1 Waste Stream Sampling and Analysis

3.4.1.1 Field Sampling of Waste Stream Soil

Soils used in the treatability test were obtained by EG&G RFP and followed the Field Sampling Plan for Sampling Plutonium-Contaminated Soils to Support Treatability Tests. This Field Sampling Plan (FSP) for soils containing plutonium from OU2 at the RFP described the sampling objectives, the location, the number of samples to be collected, and referenced the procedures for collecting the samples. Following sample collection, the drums were shipped to the Lockheed Soil Treatability Laboratory in Las Vegas, Nevada, following chain of custody procedures.

The FSP identifies four different sampling locations based on the soil type and plutonium levels. The decision was made to conduct tests on one soil sample. A soil class was selected based on its concentration of plutonium.

Soil with an approximate activity of 83 pCi $^{239+240}$Pu/g was selected. This was in accordance with previous characterization conducted at RFP (I. Litaor, 1993). Sampling procedures were outlined in the FSP, Sections 3.1 and 3.2.

A total of eight 55 gallon drums of soil were collected during two separate sampling events and shipped, according to DOT regulations, to the Lockheed Environmental facilities for characterization and treatability testing.

3.4.1.2 Radiological Analysis of Waste Stream

Initial radiological analysis of the soil delivered to LESAT-TAD was performed by the Lockheed Analytical Lab (LAL) in Las Vegas, Nevada. Two 4-g samples were split from the LES&T-CS split (first batch) for radiological analysis and submitted to LAL on May 7, 1993. Bulk sample homogenization and bulk soil sampling, described in Sections 3.4.2.2 and 3.4.2.3, respectively, were carried out prior to removal of the radiological analysis samples.

Gross alpha/beta was determined by LAL procedure LAL-91-SOP-0061, "Gross Alpha/Beta on Solid Samples."

Plutonium isotopic analysis (^{238}Pu, $^{239, 240}Pu$) was analyzed by alpha spectroscopy using LAL-91-SOP-0108. Samples were dissolved prior to assay.

^{241}Am was analyzed by alpha spectroscopy using LAL-91-SOP-0108. Samples were dissolved prior to assay.

A second set of samples were submitted to LAL on May 13, 1993, for other analyses; plutonium isotopic analysis was also performed on these samples.

One sample removed from the IT Corp. (ITC) split (first batch) was submitted to ITC, Richland, Washington on June 2, 1993, for gross alpha, gross beta, and ^{241}Am analysis.

Additionally, two samples taken from the ITC split (second batch) were submitted to ITC, Kingston, Tennessee on July 27, 1993, for ^{238}Pu, $^{239, 240}Pu$ isotopic analysis, ^{241}Am, and $^{233, 234}U$, ^{235}U, ^{238}U isotopic analysis.

The procedures used for radiological analysis by ITC and TMA Norcal were not available for this report.

Results for LAL, ITC, and TMA Norcal analyses are presented in Section 4.1.1 and Appendix A.

3.4.1.3 VOC, SVOC, and TAL Metals Analysis of Waste Stream

Two samples removed from the LES&T-CS split (first batch) were submitted to LAL for CLP volatiles and semi-volatiles analyses on May 13, 1993. Standard CLP methods were used for analyses.

One sample removed from the ITC split (first batch) was submitted to IT Corp., St. Louis, Missouri, for CLP volatiles, semi-volatiles, and TAL metals on June 2, 1993.

One sample was removed from the ITC split (second batch) and submitted to ITC St. Louis, Missouri for CLP volatiles, semi-volatiles, and TAL metals on July 30, 1993.

3.4.2 Sample Receipt, Homogenization, and Sampling

3.4.2.1 Sample Receipt and Radiological Surveys

Samples of Rocky Flats soils were received in two separate shipments. The first shipment consisted of five sealed 55-gallon drums which were received on March 31, 1993. Barrel and sample liner integrity were found to be in order, and the chain of custody seals were found intact. The barrel numbers and manifest masses were: D-81444 (184 kg), D-81445 (212 kg), D-81446 (148 kg), D-81447 (139 kg), and D-81448 (158 kg).

Radiological swiping and pancake meter surveys were conducted on the exterior of the barrels. No evidence of leakage or surface contamination WAS found. Lockheed Chain of Custody seals were then applied to the outer lock ring of each barrel; barrels were released for homogenization and sampling on May 5, 1993.

The second shipment of Rocky Flats soils consisted of three 55-gallon drums and was received on June 18, 1993. Again, barrel and sample liner integrities were found to be in order and chain of custody seals were found intact. The barrel numbers and manifest masses were: D-82208 (194 kg), D-82209 (172 kg), and D-82210 (197 kg).

No evidence of leakage or surface contamination was found by radiological swiping and pancake meter surveys. Lockheed Chain of Custody seals were attached to the barrels. Samples were then released for homogenization and sampling on June 21, 1993.

3.4.2.2 Sample Homogenization

Bulk homogenization of the first shipment of Rocky Flats soil was conducted as specified by Subsection 7.3.3, Characterization of Test Soil, of the Work Plan. This procedure followed the method ASTM C702-87, Standard Practice for Reducing Field Samples of Aggregate to Testing Size (also known as the cone and quarter method).

The first shipment of Rocky Flats soils was found to have absorbent, which was loosely placed between paper and cloth material, spread out into the soil. This condition necessitated the removal of soil which was visibly mixed with absorbent. Mixed absorbent/soil material which was removed weighed 167 kilograms (moist).

The remainder of soil was then mixed according to the homogenization method. Briefly, this method consisted of piling the contents of the barrels into a cone, with each shovelful being placed over the center of the cone. Once completely piled, the material was then spread outward from the center of the pile into a circular layer so that it was from five to ten centimeters thick. The material was then split into quarters, with one pair of opposite quarters being removed and placed back into the barrels. The remaining material was then shoveled back into a cone, taking consecutive shovelfuls from each opposing quarter.

Material was coned and quartered again four additional times, which reduced the total mass for the fifth sequence to approximately 29 kilograms. Mass of material for each sequence is recorded in Appendix A. From the fifth sequence, opposite quarters separated out for shipping to ITC for independent analysis weighed 13 kg; material remaining for the LES&T characterization sample (LES&T-CS) weighed 15.5 kg.

Due to the low mass of soil remaining for the LES&T-CS, a second series of coning and quartering was conducted to yield a large enough sample size. Material was removed from the barrels and coned and quartered for five sequences. Before the fifth sequence, the samples previously

removed for ITC and for the LES&T-CS were added back in. This composite pile was then shoveled over on itself, coned, and quartered. The sample for ITC weighed 12 kg, and the sample for the LES&T-CS weighed 38 kg.

The LES&T-CS was used for gross alpha, gross beta, plutonium, americium, VOC and SVOC, bulk density, percent air-dry moisture, pH, particle size distribution, and dense liquid separation determinations, and for Phase 1 tumbler testing and attrition scrubber tests. Grab samples from the ITC split were taken and submitted for analysis for gross alpha, gross beta, americium, plutonium, TAL metals, VOC and SVOC.

For Phase 2 testing, a second shipment of soil was necessary. Once received, this sample was also coned, quartered, and split according to the homogenization procedure, adding in the unused soil (other than the remaining ITC sample and the LES&T-CS) from the first shipment. Each sequence split was placed into separate barrels.

3.4.2.3 Bulk Sampling

Sampling of the bulk samples was performed as specified by Subsection 7.3.3, Characterization of Test Soil, of the Work Plan. This procedure was performed in conjunction with sample homogenization to generate the LES&T-CS and ITC samples.

3.4.3 Treatment Process Sampling and Analysis

3.4.3.1 Characterization Phase Sampling and Analysis

Sampling of Phase 1 characterization materials was conducted as specified by Subsection 7.3.3.4, Particle Size Analysis. Two sample splitters were used; one had an opening of 63.5 mm and the other an opening of 25.4 mm. Material which was less than 13 mm (half the opening of the chutes of the smaller splitter) was split in the smaller splitter; all other material was split in the larger splitter.

For samples in which grass obstructed the chute openings, the grass was pushed into the opening which it obstructed. This necessitated breaking up some grass which covered two or more openings.

If the whole sample volume from a characterization operation was less than the volume of a Standard Radiological Counting Container (SRCC), about 300 cm^3, it was placed into the SRCC without splitting. These samples were then analyzed for mass and activity content. All activity analysis was by gamma spectroscopy.

3.4.3.2 Phase 1 Treatment Sampling and Analysis

Sampling of Phase 1 testing materials (bench tumbler and attrition scrubber) was conducted as specified by SOP AWC 101, Soil Sampling for Radiological Analysis. Sampling in this manner may have varied from that as specified in Subsection 7.3.3 of the Work Plan. Samples in which

the whole volume was less than the SRCC volume were placed into the SRCC without being split. All activity analysis was by gamma spectroscopy.

3.4.3.3 Phase 2 Treatment Sampling and Analysis

Sampling of Phase 2 testing process products was conducted as specified by Work Plan Subsection 7.3.6 or as specified by SOP AWC 101. The use of SOP AWC 101 sampling methods may represent a variance from the Work Plan. Under SOP AWC 101 procedure, process products were thoroughly homogenized followed by the removal of two samples from opposite regions of the process product. When it was noted that this procedure varied from the Work Plan, the procedure specified by Subsection 7.3.6 was conducted on Run #7 & 8 products. A comparison of these samples to the samples removed under SOP AWC 101 is found in Section 4.1.2B. All activity analysis was by gamma spectroscopy.

3.4.3.4 Post-Phase 2 VOC, SVOC, TAL Metals, and Radiological Analyses

No post-Phase 2 process samples were analyzed for VOCs, SVOCs, or TAL metals. Two batches of process samples were submitted to TMA Norcal for independent radiological analysis. Results of these samples are discussed in Section 4.1.1. All other samples collected were analyzed for weight and by gamma spectroscopy and were archived until shipment to RFP in early 1994.

3.5 Data Management

All data and information generated throughout the treatability study were kept as project records. These records include project logbooks, laboratory notebooks, sample activity logs/MCA data sheets, sample coordinating bench sheets, Chain of Custody forms, Sample Mass Data sheets, Characterization Run Sheets, Process Equipment Run Sheets, LAL analysis results forms, Radioactive Shipping Reports, Final Sample Shipping Report, Nonconformance Reports (NCRs), Corrective Action Reports (CARs), and spreadsheet calculation forms. The purpose for maintaining these records was to permit independent verification of the results and the conclusions drawn. NCRs and CARs are discussed separately in Section 4.2.5.

3.5.1 Collection of Data From Characterization and Treatment Phases

To maintain records on the use and movement of samples, Sample Coordinating Bench Sheets were compiled. To ensure the integrity of samples which must be placed into or are moved through an unsecured area, Chain of Custody forms were maintained and seals applied to sample containers.

The various daily operations affecting the project and project specific actions, such as, testing, analyses, and general observations, were recorded in project logbooks and laboratory notebooks.

Each procedure conducted for the project in both the characterization phase and the two treatability phases had a procedure specific run sheet (characterization

run sheets or process equipment run sheets) maintained for procedure related measurements, settings, observations, and results.

In order that a separate record be maintained for weighings and for activity analysis by gamma spectroscopy, Sample Mass Data Sheets, recording gross mass data, and Sample Activity Logs/MCA Data Sheets were kept as records of gross data results, respectively.

Samples moved off-site for shipping to other labs or to be returned to the generator were accompanied by a Radioactivity Shipping Report, as required by LESAT-TAD SOPs.

Samples collected together and shipped back to RFP were listed in the Final Sample Shipping Report.

Samples analyzed by LAL for CLP SVOCs and VOCs, and for radiological analysis, generated results forms listing the findings of those analyses.

The above listed records constitute the data collection, oversight, and compliance required portions of the project records. Originals or legible copies of these records, other than laboratory notebooks, are contained in Appendix A.

3.5.2 Processing of Data

The processing of the Rocky Flats project data into information which is useable by parties tasked with oversight of activities at Rocky Flats involved the use of a MS-DOS based computerized spreadsheet program, Quattro.

Data from characterization results and from treatability test results were entered into the spreadsheet and verified for accuracy. Appropriate calculations were then entered to process the data into information. This information was then either tabularized or was graphed into useful figures. Tables were constructed in either Quattro or WordPerfect; figures were compiled in Quattro, AutoCad, or Lotus 123 for Windows.

Copies of the project spreadsheet constitutes the processed data portion of the project records and are found in Appendix A. Sample calculations are also listed in Appendix A.

3.6 Deviations from the Work Plan

Several deviations from the Work Plan occurred during testing. Some of the deviations were necessitated by operational difficulties encountered prior to or during an activity. Other deviations occurred due to the following of LESAT procedure rather than Work Plan procedure.

Section 7.3.6 of the Work Plan specifies that situations where conflicts between LESAT internal procedures and the Work Plan occur, the Work Plan takes precedence unless resolved in writing by EG&G Rocky Flats or its designated subcontractor. Deviations which were resolved prior to the activity are noted below.

1. Section 7.3.3 of the Work Plan requires about 1450 kg of soil to be coned and quartered as per the technique specified.

   During transport, a substantial volume of the first shipment of soil contained soil which had become mixed with the moisture absorbent material placed between layers of soil. As a result, only a portion of the soil was useable and two rounds of homogenization on the first shipment, and a second shipment of soil was necessary.

   The EG&G Rocky Flats Project Manager was notified of the problem of sample mixed with moisture absorbent at the time it was discovered. EG&G Rocky Flats verbally approved the procedures used to accommodate this variation, and arranged for a second shipment of soil to make up for the sample mass discrepancy.

2. Section 7.3.3 of the Work Plan requires about 22 to 23 kg of soil to be left in each of the quarters.

   More soil than this was anticipated to be used through Phase 1 testing. Splitting the sample to achieve this goal necessitated a quartering using acute and obtuse angles on the fifth split. The result was a 38 kg sample for the LES&T-CS and a 12 kg sample for ITC.

   EG&G Rocky Flats was notified that a larger sample size was anticipated prior to the second homogenization of the first batch. Verbal approval was given to use this method to retrieve the larger sample.

3. Section 7.3.3.4 of the Work Plan requires dry sieving work to use the sieves separately.

   For time effectiveness, sets of three to five sieves were nested and placed on the sieve shaker. Each set was sieved for the length of time specified in the procedure. To accommodate the sample size, several samples of soil were sieved separately. Each specific fraction (e.g., all 6.3 mm) was recombined and then homogenized prior to subsampling and radiological analysis.

   EG&G Rocky Flats was notified that a time savings can be made by nesting several sieves together on the sieve shaker. Verbal approval was given to proceed with this method.

4. Section 7.3.3.4 of the Work Plan requires the chute on the sample splitter to be opened to a minimum of twice the largest particle size.

   The largest diameter chute available at the time of this work had a maximum 6.4 cm chute opening. The largest diameter particles delivered to LESAT were just under 5.1 cm, necessitating a 10 cm chute opening.

5. Section 7.3.3.4 of the Work Plan requires each pass through the splitter to reduce the sample fraction in half.

   For the >37.5 mm sample, gravel and grass separated from each other and made operating the splitter difficult. Separate splitting of gravel and of grass was

performed. Additionally, the grass obstructed the chutes. It was decided to push the grass through the chute over which it settled, breaking apart grass which lay over 2 or more chutes.

EG&G Rocky Flats was notified of the change in procedure, prior to the work, for separately splitting the gravel and the grass in the >37.5 material and for breaking apart grass which obstructed two or more chutes. Verbal approval was given to proceed with this change.

6. Section 7.3.3.4 of the Work Plan and LESAT procedure AP TAD-3 required wet sieving samples in a nest and through the sieves unsubmerged.

Previous experience with wet sieving has found that wet sieving the 2.0 mm to 0.106 mm fractions in "pan baths" (submerged) was an effective and time/water saving means for sieving.

7. Sections 7.3.3.6, 7.3.3.7, 7.3.3.8, 7.3.4.2, and 7.3.6 of the Work Plan required performing various chemical, physical, and radiological analyses on samples generated throughout the project.

Funding provisions were not of an amount which could meet these requirements for all samples specified by the Work Plan. Samples were archived and remain available for further chemical, physical, and radiological analyses.

8. Section 7.3.4.1 of the Work Plan required particles larger than 19 mm to be removed prior to adding into the feed hopper.

The EG&G Project Manager orally specified that this size cut-off be lowered to a 6.3 mm upper size limit.

9. Section 7.3.4.1 of the Work Plan required containers holding process run products to be weighed following the Phase 2 test run. Additionally, Section 7.3.6 of the Work Plan required the use of a Tyler Sample Reducer for splitting process run slurries if the supernatant were clear enough. Process samples which could not be split immediately in the splitter were to be agitated at high speeds with a portable agitator for at least 15 minutes and then poured into a 16:1 sample reducer.

Project employees erroneously followed the method specified in SOP AWC 101 for sampling. Duplicate samples were taken from process products. When the deviation was discovered, a resampling of several process products was conducted in accordance with the procedure outlined in Sections 7.3.4.1 and 7.3.6 of the Work Plan. Further discussions of the comparison of the two methods are given in Sections 3.1.2B, 3.2.1B, 3.4.3.3, and 4.1.2B.

10. Section 7.3.4.2 of the Work Plan required samples to be removed from the overflow of the gravity separator (mineral jig) for analysis.

During the process runs, the gravity separator (mineral jig) was set-up for direct feed into the spiral classifier. All process products produced from the spiral classifier or the thickener were sampled, producing a complete mass and activity

balance for the gravity separator overflow stream. It was decided to forgo sampling at the gravity separator overflow.

4.0 Phase 2 Treatment Sampling and Analysis Results and Discussion

4.1 Data Analysis and Interpretation

The following sections provide discussions of the results of all phases of the Plutonium in Soils Treatability Study. Discussions are segmented into separate sections on waste stream characteristics, pre-Phase 1 characterization data, Phase 1 treatability testing data, and Phase 2 treatability testing data. Sections providing costing, schedule, and key contacts information follows the Phase 2 section.

4.1.1 Data Analysis of Waste Stream Characteristics

From the as-received bulk soil, samples were taken for radiological analysis, CLP volatile and semi-volatile organic compounds (VOCs, SVOCs) analysis, and target analytic list (TAL) metals. Seven samples were analyzed for ^{241}Am, five samples for ^{238}Pu and ^{239,240}Pu, three samples for gross alpha and gross beta, two samples for ^{233,234}U, ^{235}U, and ^{238}U, three samples for VOC/SVOCs, and two samples for TAL metals.

4.1.1.1 Radiological Analysis

A total of nine samples were analyzed for various radiological parameters and radionuclides. The results of these analyses are presented in Figure 4.1.

Though close agreement exists for several samples, some variability between values for each of the radiological measurements does occur (e.g., from 7.9 to 17.88 pCi ^{241}Am/g soil). The reason for the variance in these values is unclear.

^{241}Am was used throughout the study as a measure for the presence of ^{239,240}Pu, which itself was used as a single indicator constituent for gross alpha, gross beta, and ^{238}Pu. The establishment of the ratio of ^{239,240}Pu to ^{241}Am was significant for evaluating the performance of the TRUclean process.

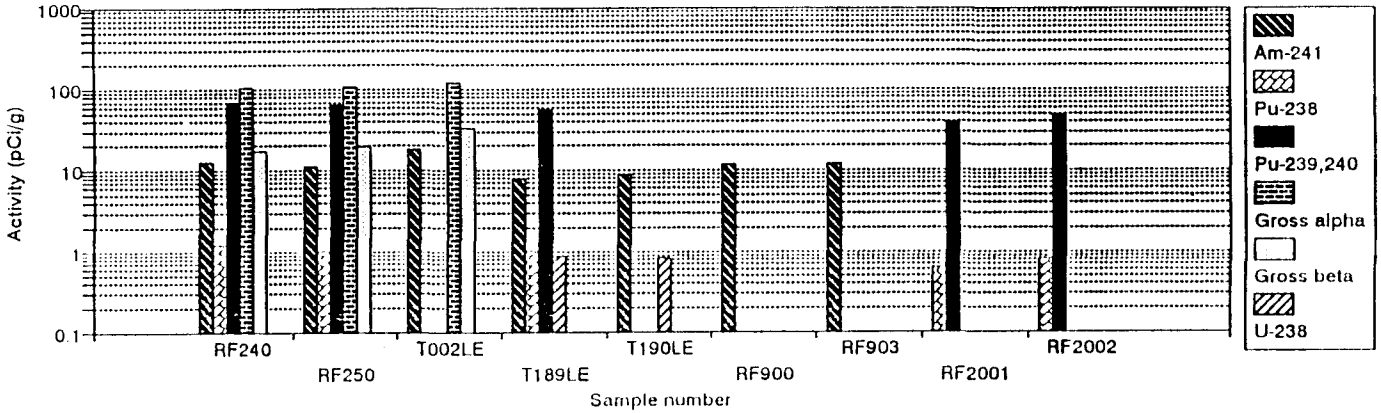
Results from the analysis of three samples for ^{241}Am, ^{239,240}Pu, and the ratio of ^{239,240}Pu to ^{241}Am are presented in Table 4.1 and graphed in Figure 4.2. The reason for the variance in ratios between sample T189LE and samples RF240, RF250 is unclear.

Sample ID	^{241}Am Activity (pCi/g)	^{239,240}Pu Activity (pCi/g)	$\frac{^{239,240}\text{Pu}}{^{241}\text{Am}}$
RF240	12.8 ±0.52	71.5 ±2.6	5.586
RF250	11.2 ±0.47	64.6 ±2.4	5.768
T189LE	7.9 ±1.68	57.1 ±11.9	7.228

Table 4.1 Comparative Results for $^{239/240}$Pu/^{241}Am Ratio

RADIOLOGICAL RESULTS, BULK SOIL

Rocky Flats, OU2



Sample	Am-241	Pu-238	Pu-239, 240	Gross A	Gross B	U-233, 234	U-235	U-238
RF240	12.8 ±0.52	1.202 ±0.092	71.5 ±2.6	108 ±16	17.3 ±4.6	———	———	———
RF250	11.2 ±0.47	1.02 ±0.083	64.6 ±2.4	110 ±16	19.7 ±4.6	———	———	———
T002LE	17.88 ±1.76	———	———	123.1 ±23.1	32.89 ±3.69	———	———	———
T189LE	7.9 ±1.66	1.05 ±0.39	57.1 ±11.9	———	———	0.685 ±1.76	0.0222 ±0.204	0.876 ±0.216
T190LE	8.84 ±1.87	———	———	———	———	0.724 ±0.22, 0.185	0.0382 ±0.412, 0.248	0.8445 ±0.26, 0.198
RF900	11.77 ±0.461	———	———	———	———	———	———	———
RF903	11.87 ±0.865	———	———	———	———	———	———	———
RF2001	———	0.626 ±0.066	38.7 ±1.5	———	———	———	———	———
RF2002	———	0.819 ±0.075	48.4 ±1.8	———	———	———	———	———

Figure 4.1 Radiological Analysis Results of Waste Stream (bulk) Soil. All values are in pCi/g.

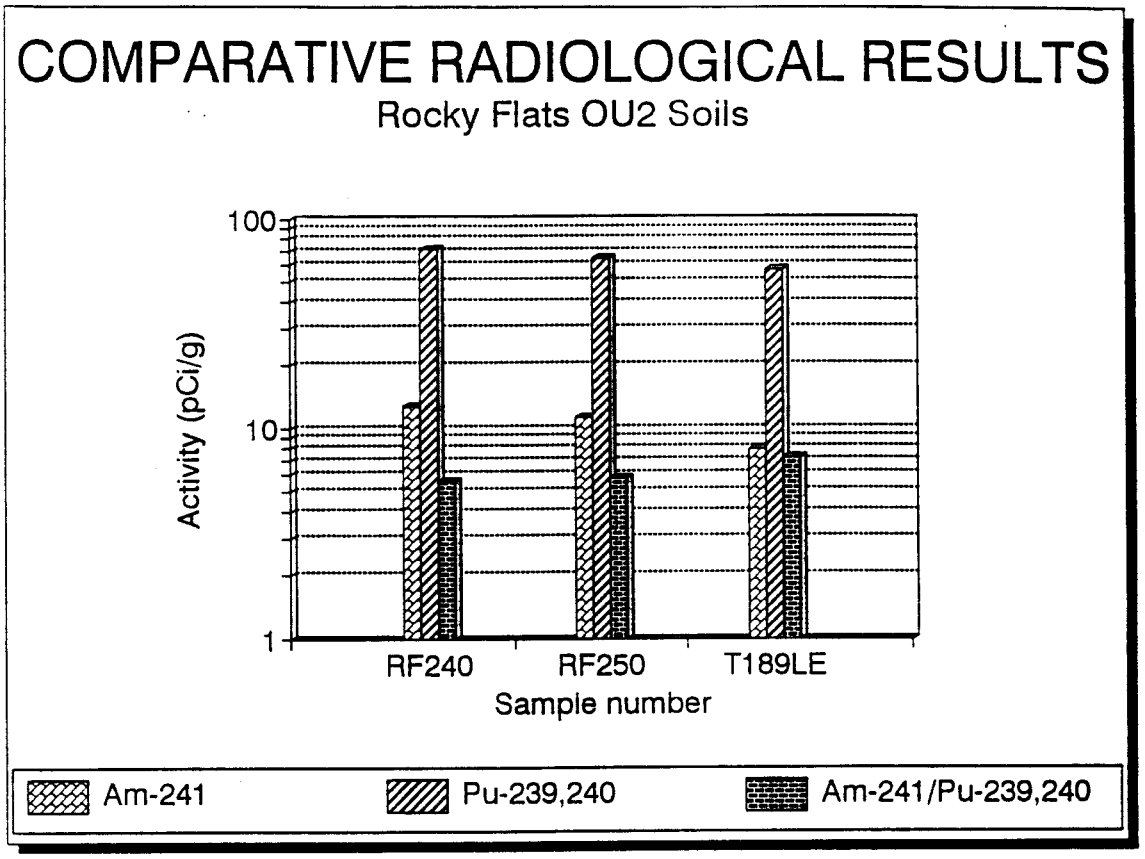


Figure 4.2 Comparative Results for $^{239, 240}Pu/^{241}Am$ Ratio

Further discussions of $^{239,240}Pu$ and gross alpha/gross beta analyses are given in Section 4.1.2B.

4.1.1.2 Volatile and Semi-Volatile Organic Compounds Analysis

To establish the presence and level of CLP VOCs and SVOCs, three samples were submitted to CLP laboratories for analysis.

Results of these analyses are contained in Appendix A.

4.1.1.3 TAL Metals Analysis

To establish the presence and level of TAL metals, two samples were submitted to CLP laboratories for analysis.

Results of these analyses are contained in Appendix A. The background levels of these metals for this soil is unknown and a comparison cannot be made.

4.1.2 Data Analysis of Characterization Data

The results of several analyses for various soil properties is discussed in the following subsections. Each technique is discussed separately.

4.1.2 A Primary Methods

4.1.2.1 Bulk Density Results & Discussion

Measured bulk density values are listed in Table 4.2. The average value of 1.02 g/cm^3 is within the expected range for a developed soil which contains an estimated minimum of 1% by weight organic matter.

Table 4.2 Bulk Density

Replicate #	Volume (cm^3)	Dry Mass (g)	Bulk Density (g/cm^3)	Percent H_2O
RF-1	140	148.33	1.06	11.2
RF-2	140	138.11	0.99	11.7

Large gravel (>19 mm) did not visibly appear in the bulk density samples. As this material makes up at least 8% of the less-than 50 mm diameter soil, some variation from this value can be expected in additional samples.

4.1.2.2 Percent Air-Dry Moisture Determination Results & Discussion

The results for percent moisture content are listed in Table 4.3, on an oven dry basis.

Table 4.3 Percent Moisture Content

Replicate #	Dry Net Mass (g)	Water Mass (g)	Percent Moisture
RF-3	125.173	2.882	2.30
RF-4	132.284	2.852	2.16

With an average value of 2.23%, considerably less moisture was present in the air dry sample used for Phase 1 testing than that of the soil used for Phase 2 testing (average of 11.5%). Though the soil attains a relatively low air dry moisture content under the desiccating conditions of the lab, the water holding capacity, and thus the saturation water content, can be expected to be quite high due to the high content of clay and silt minerals and organic matter.

4.1.2.3 pH Determination Results & Discussion

Measured values, averages, and standard deviations for pH determinations are presented in Table 4.4. All measurements were obtained within one hour; room temperature remained at 24°C during this period.

Table 4.4 Soil pH

	pH (H_2O)	pH (0.01M $CaCl_2$)
Replicate 1	7.61	7.12
Replicate 2	7.63	7.14
Replicate 3	7.60	7.14
Average	7.61	7.13
Std. dev.	0.015	0.012

The average pH value of 7.61 obtained from suspensions in water is classified as slightly alkaline (Donahue, 1977, Fig. 5-7), indicative of soils in dry climates. The average pH value of 7.14 obtained from suspensions in 0.01 M calcium chloride is classified as very slightly alkaline, just to the basic side of neutral. The lower pH values for the 0.01 M $CaCl_2$ suspensions is expected as calcium ions are thought to exchange for surface protons (Koehler, et al., 1986).

Without a measurement of the organic and inorganic anions present in solution a prediction of the speciation of plutonium in these samples is not possible. At neutral to slightly alkaline pH values plutonium has a very low solubility in water (Cleveland, 1979). Calcium carbonate is not expected in this soil as the pH is below 7.8 (Page, et al., 1982).

4.1.2.4 Particle Size Analysis Results & Discussion

Data values for percent mass, percent activity, pCi ^{241}Am/g and calculated 239, 240Pu are depicted in Figures 4.3 and 4.4, Dry and Wet Sieve Results, respectively. These figures illustrate the sieve opening diameters upon which mass and activities were retained and the general order of

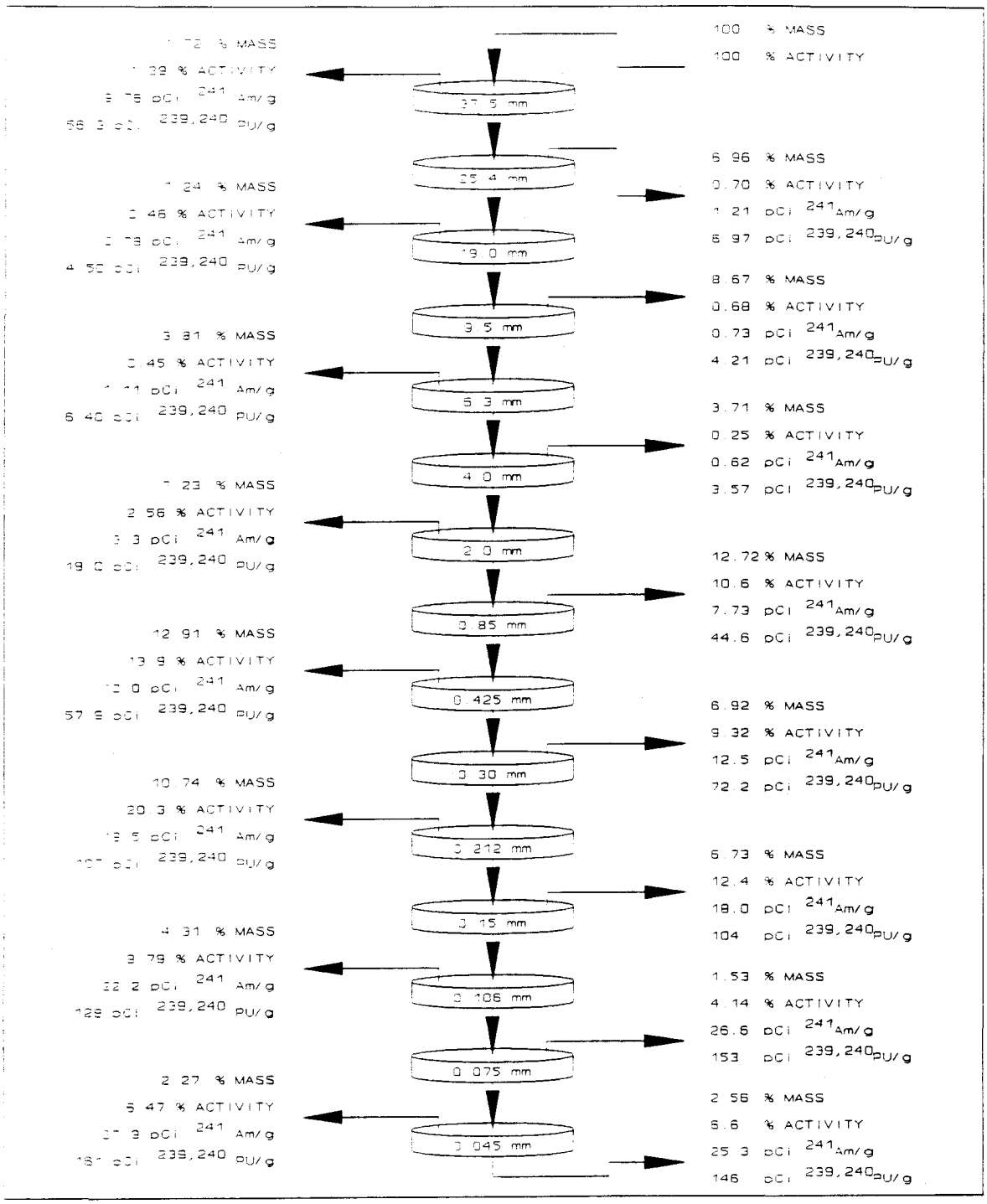


Figure 4.3 Dry Sieve Results

Size of Sieve Openings (mm/in. or mesh)	Percent Mass	Percent Activity	Measured Americium Activity ($pCi^{241}Am/g$)	Calculated Plutonium Activity ($pCi^{239, 240}Pu$)
37.5/1.5"	1.72	1.39	9.76	56.3
25.4/1.0"	6.96	0.70	1.21	6.97
19.0/0.75"	7.24	0.46	0.78	4.50
9.5/0.37"	8.67	0.68	0.73	4.21
6.3/0/25"	3.81	0.45	1.11	6.40
4.0/5 mesh	3.71	0.25	0.62	3.57
2.0/10 mesh	7.23	2.56	3.3	19.0
0.85/20 mesh	12.72	10.6	7.73	44.6
0.425/40 mesh	12.91	13.9	10.0	57.9
0.300/50 mesh	6.92	9.32	12.5	72.2
0.212/70 mesh	10.74	20.3	18.5	107.0
0.150/100 mesh	6.73	12.4	18.0	104.0
0.106/140 mesh	4.31	9.79	22.2	128.0
0.075/200 mesh	1.53	4.14	26.6	153.0
0.045/325 mesh	2.27	6.47	27.9	161.0
<0.045 mm	2.56	6.6	25.3	146.0

Dry Sieve Results.

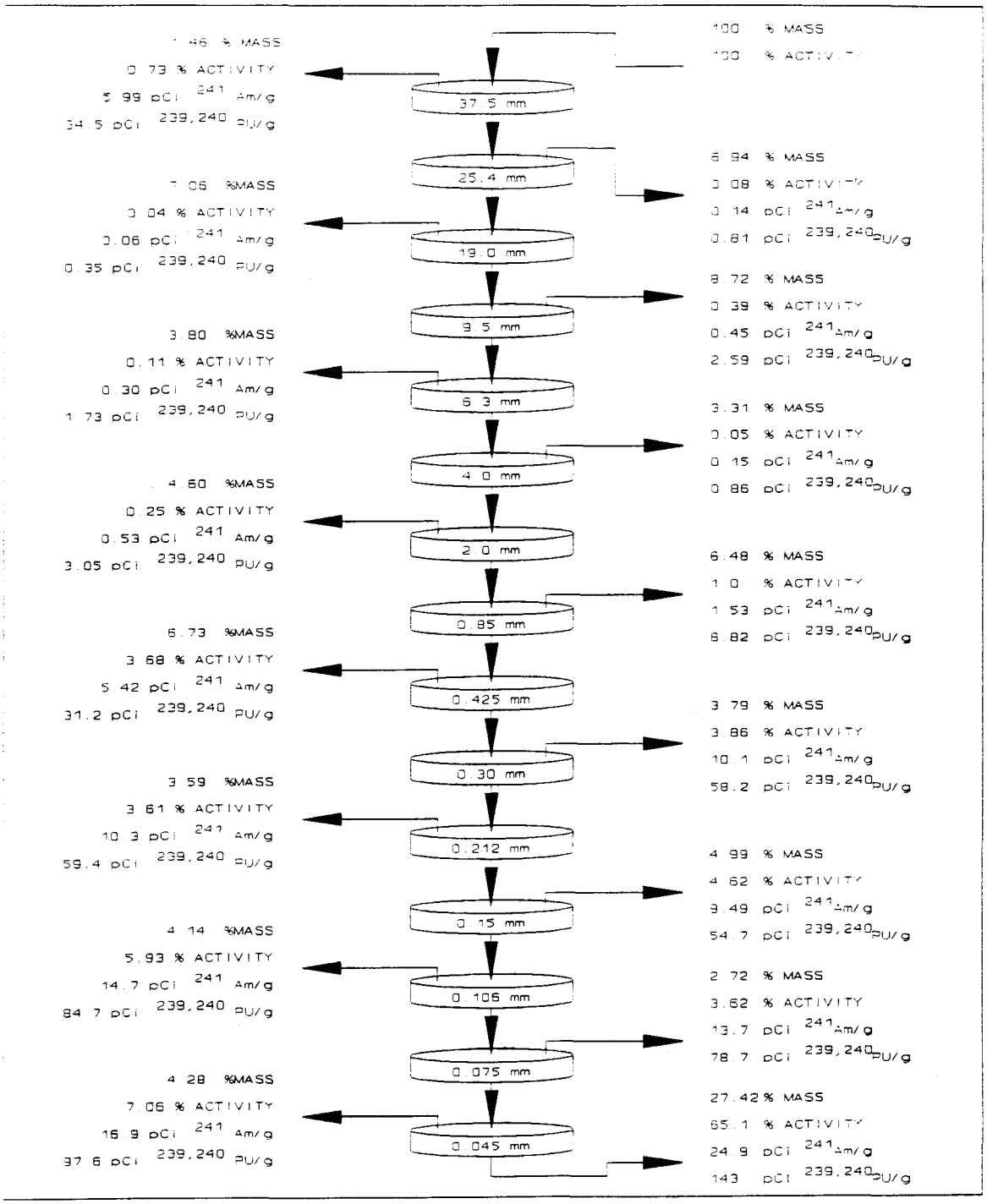


Figure 4.4 Wet Sieve Results

Size of Sieve Openings (mm/in. or mesh)	Percent Mass	Percent Activity	Measured Americium Activity ($pCi^{241}Am/g$)	Calculated Plutonium Activity ($pCi^{239,240}Pu$)
37.5/1.5"	1.46	0.73	5.99	34.5
25.4/1.0"	6.94	0.08	0.14	0.81
19.0/0.75"	7.06	0.04	0.06	0.35
9.5/0.37"	8.72	0.39	0.45	2.59
6.3/0.25"	3.80	0.11	0.30	1.73
4.0/5 mesh	3.31	0.05	0.15	0.86
2.0/10 mesh	4.60	0.25	0.53	3.05
0.85/20 mesh	6.48	1.0	1.53	8.82
0.425/40 mesh	6.73	3.68	5.42	31.2
0.300/50 mesh	3.79	3.86	10.1	58.2
0.212/70 mesh	3.59	3.61	10.3	59.4
0.150/100 mesh	4.99	4.62	9.49	54.7
0.106/140 mesh	4.14	5.93	14.7	84.7
0.075/200 mesh	2.72	3.62	13.7	78.7
0.045/325 mesh	4.28	7.06	16.9	97.6
<0.045 mm	27.42	65.1	24.9	143.0

Wet Sieve Results

the sieve sequences. The <0.045 mm opening diameter sample point indicates soil fraction smaller than 45 micron diameter.

Dry and wet sieve percent mass distributions are graphed in Figure 4.5. The most notable feature of this data is the loss of mass from middle size dry sieve fractions and subsequent gain of mass in the lower three fractions following wet sieving. A 23% increase in the less-than 45 micron diameter fraction indicates a large release of silt and clay size material from aggregates in the 150 to 2000 micron diameter size range.

Figure 4.6 depicts the ^{241}Am percent activity distribution for the dry and wet sieve fractions. Again, the loss of activity from the middle size fractions of dry sieving is complimented by the gain of activity in the less-than 45 micron diameter fraction following wet sieve. The majority of activity for this Rocky Flats OU2 soil (97.5%) lies in size fractions from the 0.425 mm opening diameter sieve and smaller, with 65% of overall activity residing in the clay and silt size fractions.

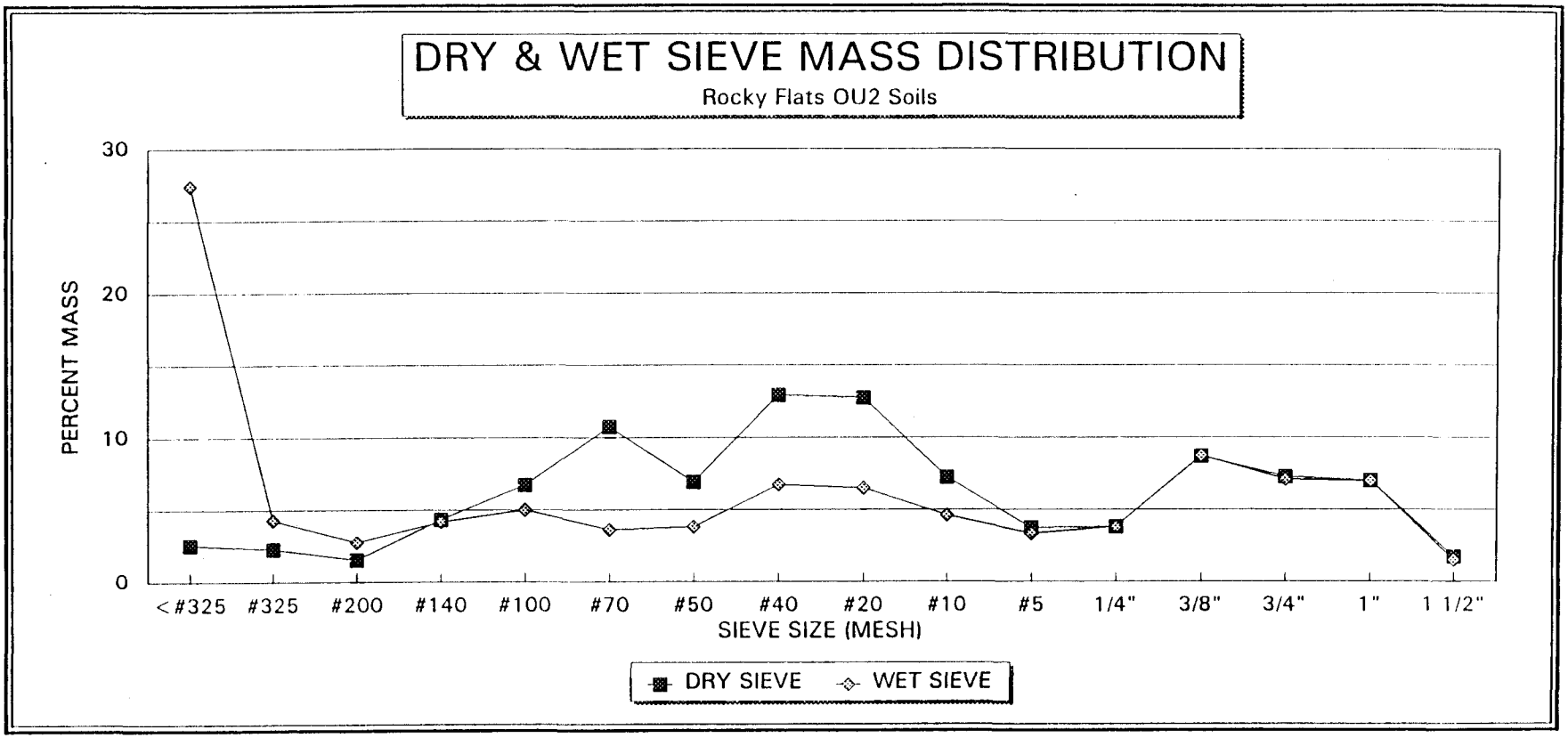
The activity to mass ratios, graphed in Figure 4.7, for both the dry and wet sieving follow the same upwards trends, with an increase in ratio with decrease in particle size diameters. Nearly all wet sieve activity ratios reported only slightly smaller values than the dry sieve fractions.

This trend, and the strong spike upwards of activity level in the less-than 45 micron diameter wet sieve ratio gives a strong indication that the plutonium resides with the clay and silt size fractions either as discrete particles or bound up to organic or inorganic particles.

The small upward trend in activity to mass ratios for the wet sieve data peaking at the 9.5 mm diameter fraction was not accompanied by any increase in observable, potentially plutonium containing, organic matter. The apparent increase in activity may be due to counting statistics. Additionally, the possible presence of larger amounts of irregular surfaces on this fraction which could hold clay and silt particles against wet sieving action could account for this increase. As americium/plutonium will not "diffuse" into solid structures it is believed to be bound up only to surfaces across the gravel faces.

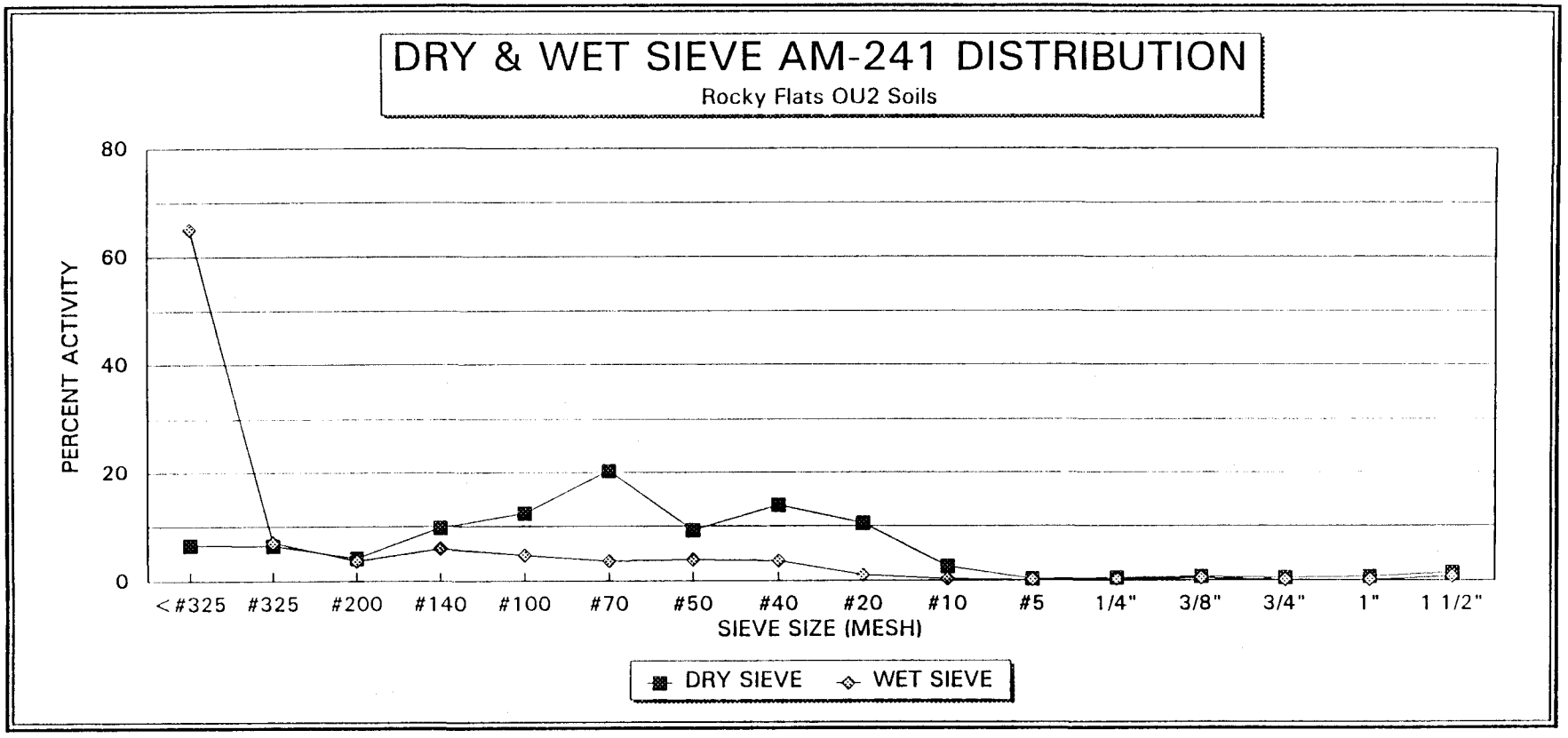
Finally, the values of 9.8 and 6.0 pCi$^{239+240}$Pu/g for the greater than 37.5 mm diameter dry and wet sieve fractions, respectively, are closely correlated to the presence of organic matter, specifically undecomposed grasses. Activity per mass values for organic matter found in this fraction, were 41.1 and 21.2 pCi$^{239+240}$Pu/g for dry and wet sieving, respectively. If the organic matter is removed from this fraction, the activity of the remaining inorganic gravel fraction falls to 0.64 and 0.56 pCi$^{239+240}$Pu/g for the dry and wet sieve, respectively.

Some residual clay and silt particles were found to remain on the grass roots despite vigorous rubbing with gloved fingers during wet sieving. While it is possible the americium/plutonium is held strictly on attached inorganic particles, other testing indicates that some other mechanism of americium/plutonium-organic matter association may have taken place.



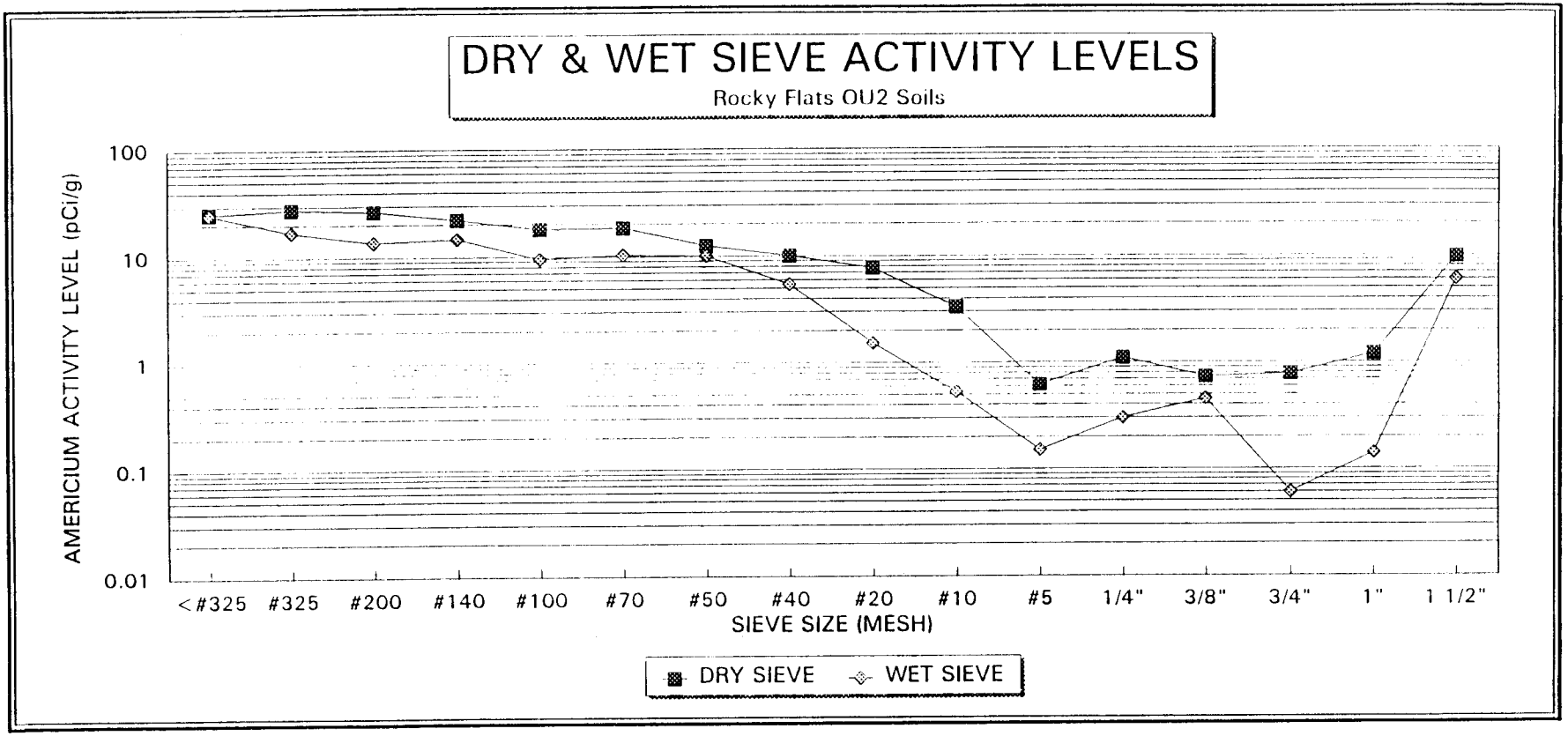
Mesh or in. Size	<#325	#325	#200	#140	#100	#70	#50	#40	#20	#10	#5	1/4"	3/8"	3/4"	1"	1 1/2"
Metric Size, mm	<0.045	0.045	0.075	0.106	0.150	0.212	0.300	0.425	0.850	2.0	4.0	6.3	9.5	19	25.4	37.5
% Mass Dry Sieve	2.6	2.3	1.5	4.3	6.7	10.7	6.9	12.9	12.7	7.2	3.7	3.8	8.7	7.2	7.0	1.7
% Mass Wet Sieve	27.4	4.3	2.7	4.1	5.0	3.6	3.8	6.7	6.5	4.6	3.3	3.8	8.7	7.1	6.9	1.5

Figure 4.5 Dry and Wet Sieve Mass Distribution



Mesh or in. Size	<#325	#325	#200	#140	#100	#70	#50	#40	#20	#10	#5	1/4"	3/8"	3/4"	1"	1 1/2"
Metric Size, mm	<0.045	0.045	0.075	0.106	0.150	0.212	0.300	0.425	0.850	2.0	4.0	6.3	9.5	19	25.4	37.5
% Activity Dry Sieve	6.6	6.5	4.1	9.8	12.4	20.3	9.3	13.9	10.6	2.6	0.3	0.5	0.7	0.5	0.7	1.4
% Activity Wet Sieve	65.1	7.1	3.6	5.9	4.6	3.6	3.9	3.7	1.0	0.3	0.1	0.1	0.4	0.0	0.1	0.7

Figure 4.6 Americium-241 Activity Distribution for Dry and Wet Sieve Fractions



Mesh or in. Size	<#325	#325	#200	#140	#100	#70	#50	#40	#20	#10	#5	1/4"	3/8"	3/4"	1"	1 1/2"
Metric Size, mm	<0.045	0.045	0.075	0.106	0.150	0.212	0.300	0.425	0.850	2.0	4.0	6.3	9.5	19	25.4	37.5
pCi Am/g Dry Sieve	25.3	27.9	26.6	22.2	18.0	18.5	12.5	10.0	7.7	3.3	0.6	1.1	0.7	0.8	1.2	9.8
pCi Am/g Wet Sieve	24.9	16.9	13.7	14.7	9.5	10.3	10.1	5.4	1.5	0.5	0.2	0.3	0.5	0.1	0.1	6.0

Figure 4.7 Americium-241 Activity to Mass Ratios for Dry and Wet Sieve Fractions

Both the observation of americium containing grass leaves separated from grass stems and roots (Section 4.1.2B, Miscellaneous Testing, Grass), and the finding of an americium containing organic matter float product from process testing runs (Section 4.1.4.3) gives a reasonable expectation that americium/plutonium could be held either intracellularly, such as in the grass leaves, or is adsorbed to outer plant surfaces. In no instance, under all analytical and processing work, was any organic material found free of activity.

While pipet analysis of smaller size fractions (Section 4.1.2B, Miscellaneous Testing, Pipet Analysis) yielded very high activity levels for the 2, 5 and 20 micron diameter size soil fractions, the separation of a float product with high activity at 1.9 g/cm^3 density of a dense liquid (Section 4.1.2.6, Dense Liquid Characterization) also indicated an association of americium/plutonium with the less dense (float product) organic matter.

4.1.2.5 Gamma Spectroscopy Analysis Results & Discussion

Results for gamma spectroscopy analysis were presented in Table 4.1 and Figure 4.2. Samples RF250, RF900, and RF903 are within two standard deviations of each other. Other sample results fall outside this group. Apparently, variability for samples taken from homogenized bulk soil does occur, most likely due to the heterogeneity of particle sizes (i.e., the presence of gravel).

Some samples in the treatability study contained significant amounts of naturally occurring radioactive materials (NORM) such as uranium and thorium. These NORM components also emit gamma photons in the region of the 60 keV americium peak which sometimes interfered with the analysis. A spectrum printout for this result was unavailable.

4.1.2.6 Dense Liquid Characterization Results & Discussion

The activity per mass values for the three float and one sink products for each replicate is graphed in Figure 4.8. The mass distribution for the replicates is depicted in Figure 4.9. The total mass for both replicates sums to greater than 50 g, indicating that not all polytungstate was washed from the samples.

Two features stand out from the graphs. First, the mass for the less-than 1.9 g/cm^3 is roughly 10% of the overall mass for the less-than 45 micron diameter fraction. As most soil minerals have densities around 2.6 g/cm^3, this would indicate a rather high organic matter content. Some very fine (<1.0 micron diameter) inorganic minerals may have been caught in the float product, but the activity to mass values are close to those found for organic material in larger size fractions. In addition, the float material is very black, and under a microscope, appears to be made up of irregular spheres. These results indicate that americium/plutonium is associated with organic matter.

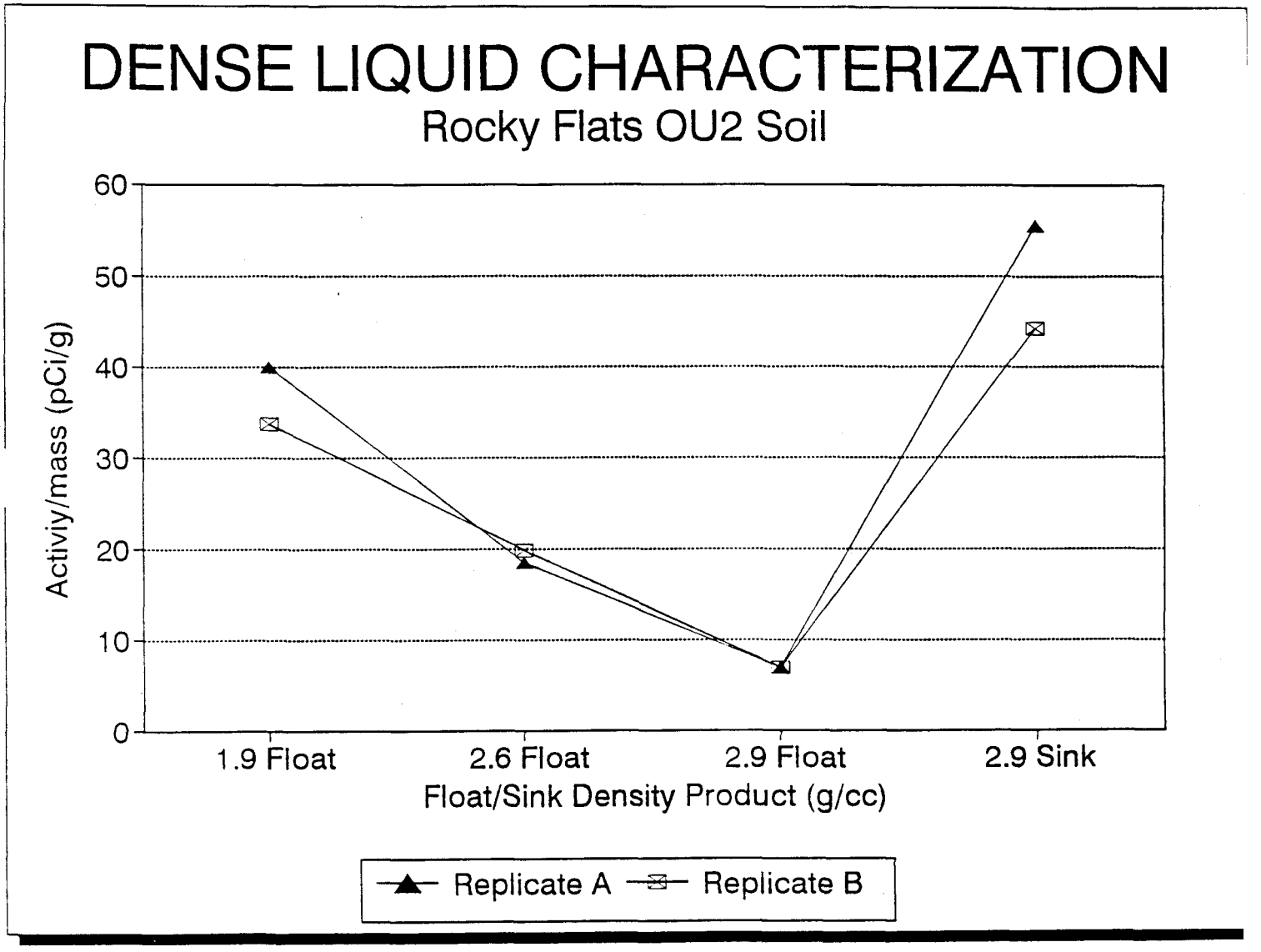


Figure 4.8 Activity Values for Dense Liquid Separated Soil (<45 micron)

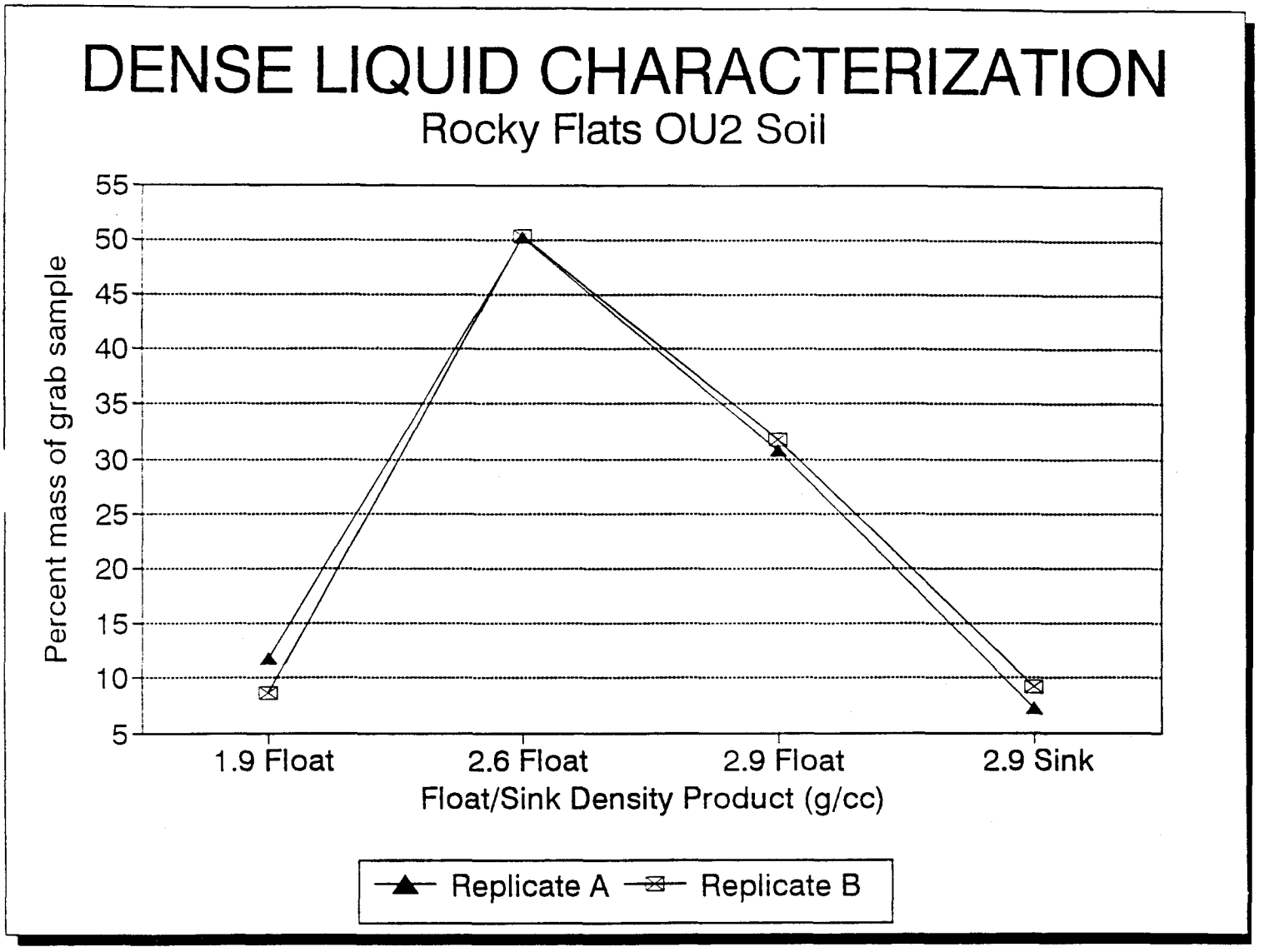


Figure 4.9 Mass Distributions for Dense Liquid Separated Soil (<45 micron)

Second, the majority of mass floats at the 2.6 g/cm^3 treatment, as expected for soil minerals, and is tan to white in color, indicative of silicate minerals. From the remaining material, however, there is roughly 8% of dense material which has activity per mass values near that for the thickener (Phase 2) product. This would indicate that density separations of americium and plutonium particles might be possible with separation out from the less dense silicate materials using techniques geared toward the less-than 45 micron diameter fraction.

4.1.2.7 Pipet-Method Analysis Results & Discussion

The results from the pipet-method analysis are graphed in Figure 4.10. Values from wet sieve fractions of 45 and 75 micron diameters were added for comparison.

As the available surface area and reactivity of the surface area increases dramatically with decreasing particle diameter, the increase in activity per mass for the smaller size fractions is not surprising. At the 2 micron diameter sample, the activity level increases to nearly 350 pCi ^{241} Am/g.

While this was not a quantitatively derived sample (no prior homogenization), the results do indicate a greater amount of americium and plutonium can be expected to exist in the clay fraction (less than 2 micron diameter) either as discrete particles or as adsorbed species. Surface speciation data has not been published for plutonium in soils; it has been speculated that plutonium may most strongly adsorb to or coprecipitate with iron and manganese oxides (Means, et al, 1978).

4.1.2 B Miscellaneous Methods

Plutonium 239 + 240 Determination Results and Discussion

The results of these analyses are shown in Table 4.5. The data indicates the

Table 4.5 Plutonium and Americium Activity of Soil Samples

Sample Number	Americium-241 Activity (pCi/g)	Plutonium-238 Activity (pCi/g)	Plutonium 239 + 240 Activity (pCi/g)
RF317240	12.83 ± 0.52	1.202 ± 0.092	71.5 ± 2.6
RF317250	11.2 ± 0.47	1.02 ± 0.47	64.6 ± 2.4
Average ratio	Plutonium 239 & 240/Americium-241 = 5.76		

ratio of $^{239, 240}$Pu to ^{241}Am was 5.76 at the time of the measurement. Since the half-life of the isotopes involved is long compared to the treatability study time frame, no additional decay corrections were applied to the established ratio.

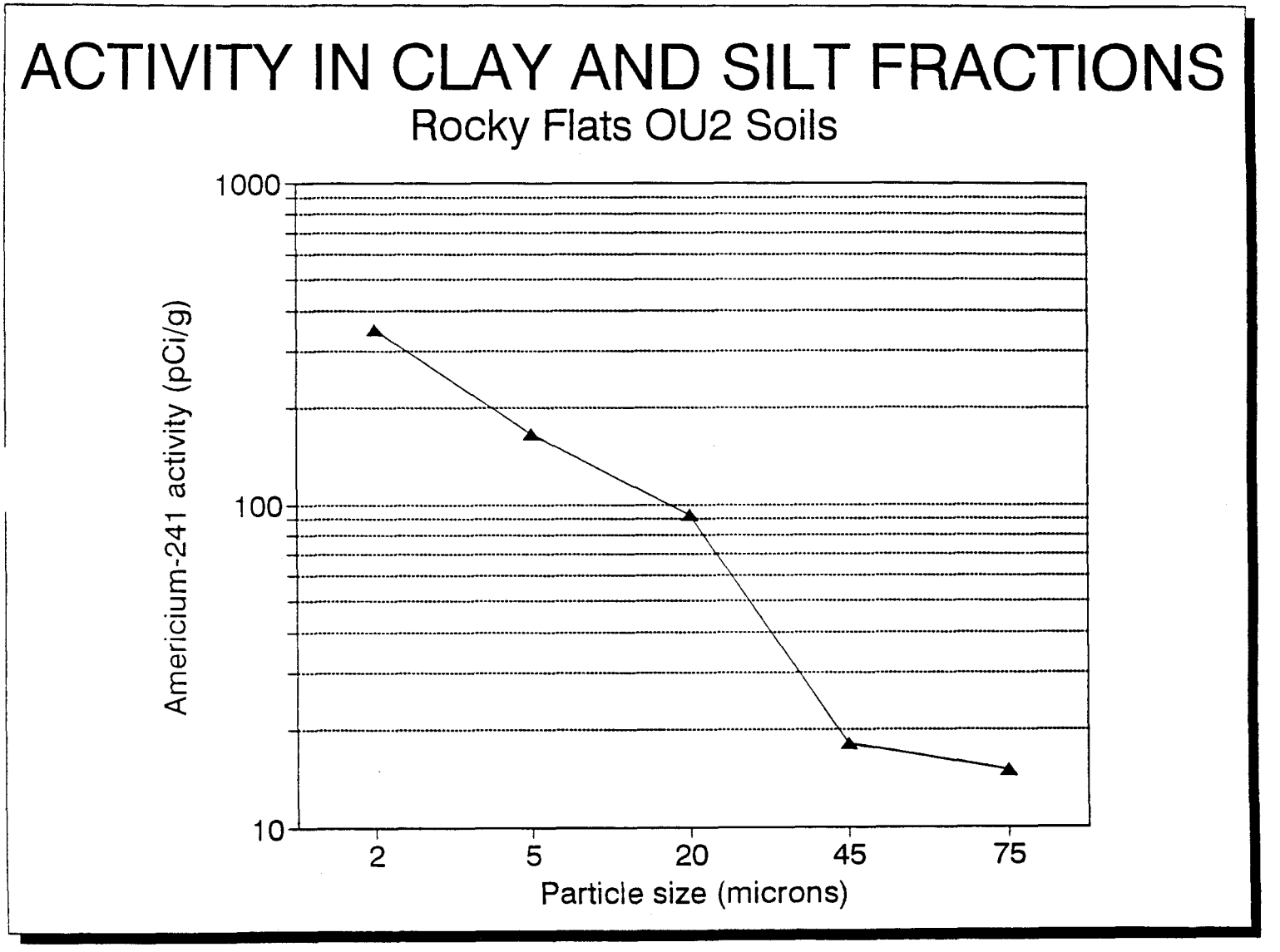


Figure 4.10 Activity Levels for Silt and Clay Fractions Separated by the Pipet Method

All activity measurements reported in the appendix spreadsheets are ^{241}Am values unless specified otherwise. To obtain $^{239, 240}Pu$ activities, the ^{241}Am values must be multiplied by 5.76.

Both americium and plutonium have a very low solubility in water. Although the solubility of americium is higher in water than plutonium, the fraction of americium which is solubilized is small compared with the total americium in the soil. Based on this observation, americium is a good indicator of plutonium in the soil/water environment. Soil samples obtained during testing and analyzed for plutonium should verify this hypothesis.

Gross Alpha and Gross Beta Determination Results and Discussion

Results of the gross alpha and gross beta analyses are shown in Table 4.6. A wide variation was found for gross alpha/^{241}Am ratios. Caution should be exercised in using gross alpha and gross beta activities as an indicator of plutonium activity since the presence of NORM components can skew conclusions drawn from the results. Selected samples from the characterization study and traceability tests were sent to outside laboratories for analysis of gross alpha and gross beta activities.

Table 4.6 Gross Alpha and Gross Beta Analysis of Soil Samples

Sample Number	Americium-241 Activity (pCi/g)	Gross Alpha Activity (pCi/g)	Gross Beta Activity (pCi/g)
RF317240	12.83 ± 0.52	108 ± 16	17.3 ± 4.6
RF317250	11.2 ± 0.47	110 ± 16	19.7 ± 4.6
Average ratio	Gross alpha/americium-241 = 9.12		
Average ratio	Gross beta/americium-241 = 1.55		

Mineralogy Analysis Results and Discussion

Qualitatively, two groups of minerals are indicated. The first group is identified by gamma peaks on several samples. These peaks indicate the presence of ^{232}Th and possibly ^{238}U. These peaks are most pronounced in the Wilfley Table Concentrate (Section 4.1.4.4) gamma spectrums. Natural uranium and thorium bearing minerals are suggested.

Also, for all fine fractions (<45 micron diameter, thickener products) widespread cracks formed upon the drying of a slurried sample. This phenomenon generally occurs when expanding clay minerals are present. The smectite group of layered clays are most responsible for swelling behavior; of this group, montmorillonite is the most commonly found smectite (Donahue, et al., 1977), and is suggested to be a member of the mineral suite.

Organic Leaves and Roots Analysis Results and Discussion

Mass and activity level results are tabularize in Table 4.7 below and graphed in Figure 4.11.

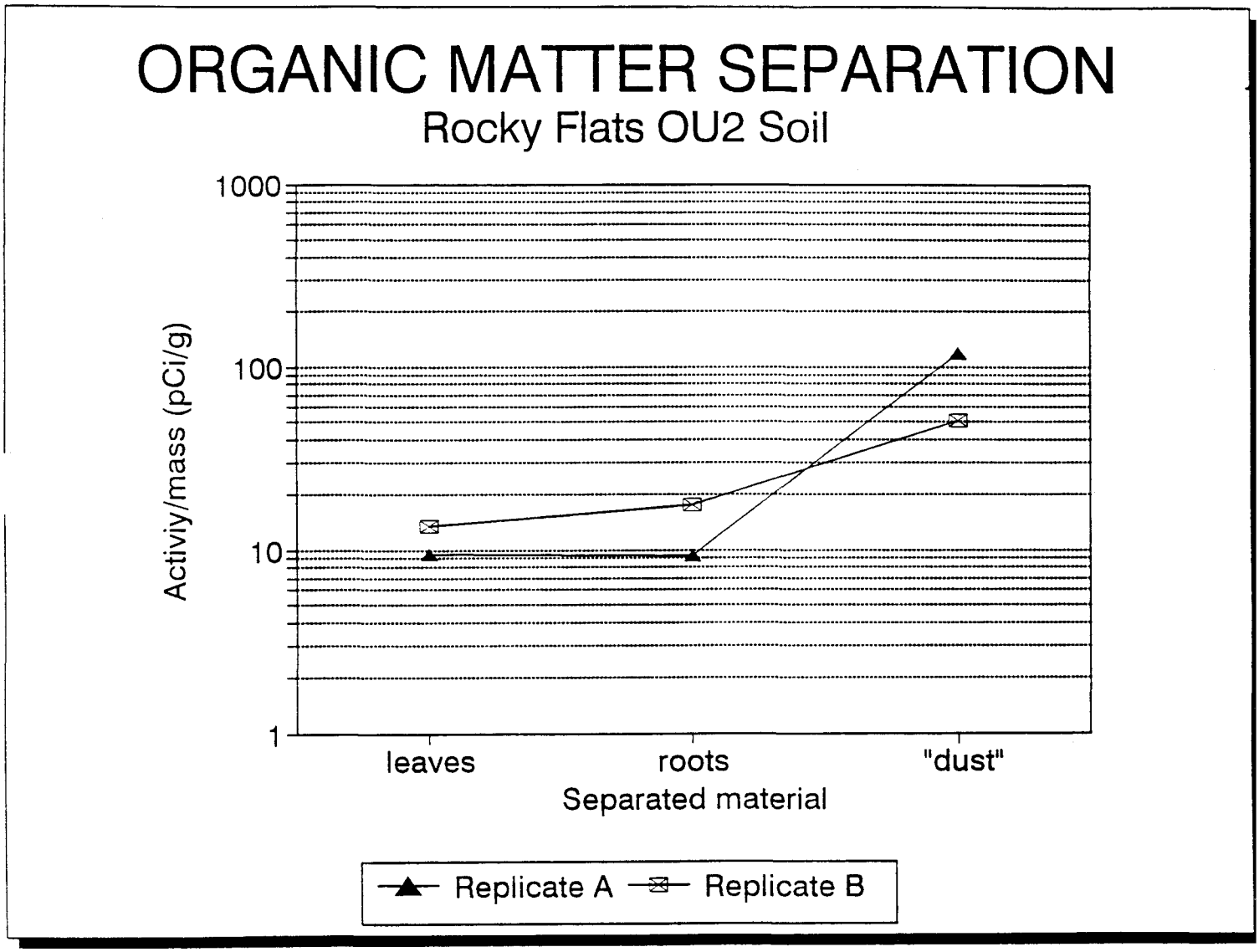


Figure 4.11 Activity Levels for Hand-Separated Fractions of Undecomposed Organic Matter

Table 4.7 Activities in Organic Fraction

	Replicate A		Replicate B	
	Mass (g)	Activity pCi ^{241} Am/g	Mass (g)	Activity pCi ^{241}Am/g
Leaves	5.332	12.50	9.184	13.55
Roots	27.657	12.01	33.379	17.72
Dust	10.589	156.66	8.187	50.67

As seen by the results, the dust material coming off of the roots and leaves is both high in activity level and quite variable. This may indicate that the source of the americium might reside in the inorganic clay and silt fractions. The similar results for the leaves and roots between both replicates could, however, suggest some other mechanism of americium-organic matter association, as mentioned previously.

Particle Surface Photography Results and Discussion

Photographs are provided in Appendix A. Photo 4.10 P-1 is of a flat >37.5 mm diameter stone which was wet sieved. Light was provided normal to the surface. Shifting the lighting to the sides reveals a highly irregular surface with pits and fractures. These surface irregularities contain silt, clay, and organic material (Photo 4.10 P-2 & P-3).

Material cemented to flat surfaces, as recorded in Photos 4.10 P-4 & P-5, may be strongly held against removal by wet sieving. The use of abrading processing techniques, such as autogenous grinding or attrition scrubbing, can readily remove surface coatings and gradually wear down raised surfaces to remove additional clays and organics in pits and fractures (Photos 4.10 P-6 & P-7, Process Run #8 treated gravel).

The length of treatment time is very important, as difficult to remove coatings will remain on some surfaces for long periods of action (Photo 4.10 P-8, Process Run #8 treated gravel).

Sampling Method Comparison Results and Discussion

The results of the original sampling under SOP AWC 101 and resampling under Work Plan Subsection 7.3.6 are graphed in Figure 4.12. These are averages for both samples from each method (except for one sample removed for thickener material under the Lockheed method). Most samples compare very closely in activity levels, with the greatest variations occurring with the Trommel Oversize Run #27 material and the +4.8 mm product.

In all but two samples (classifier clean-out, Run #8, trommel oversize Run #27), the samples taken under the Lockheed method consistently yielded higher activity levels than did the Work Plan procedure. The reason for this is unknown.

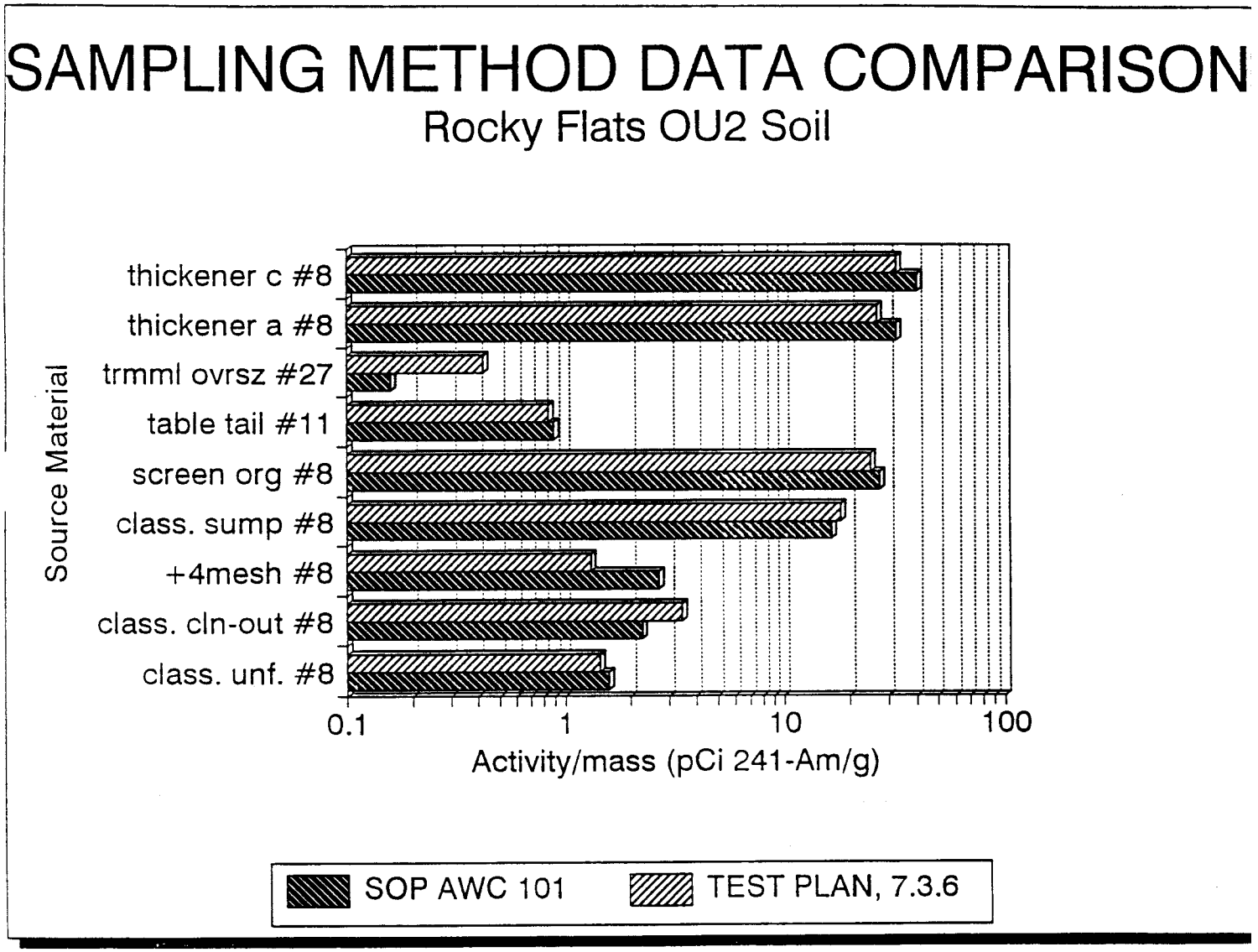


Figure 4.12 Comparison of Sampling Results
for Method SOP AWC 101 and Work Plan Subsection 7.36

Process Water Filter Test Results and Discussion

The results of the process water filter test are listed below.

Filter Paper Activity	Filtered Water Activity
pCi ^{241}Am/g	pCi ^{241}Am/L
219.11 (0.146g)	12.42 (3 L)

Approximately half the activity (32.0 pCi) was captured on the filter paper with the remaining (37.3 pCi) passing through the filter paper. Apparently some unfilterable americium exists in a sub -2.5 micron size fraction, either complexed, suspended, or dissolved.

4.1.3 Data Analysis of Phase 1 Treatability Study Data

4.1.3.1 Autogenous Grinding Results and Discussion

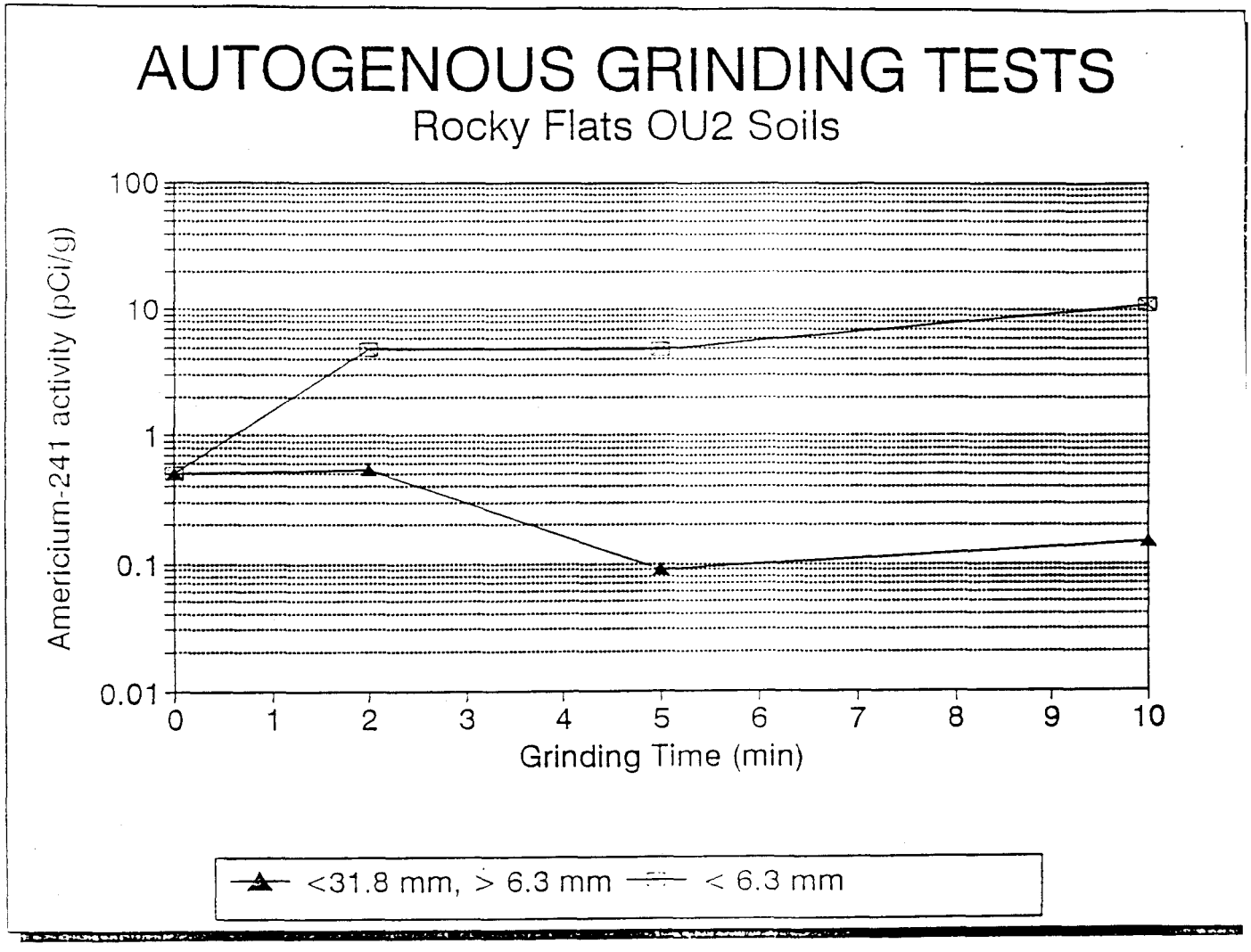
The results of this test are shown in Figure 4.13. The activity levels on >6.3 mm diameter material showed a reduction in activity following 5 minutes of autogenous grinding followed by no reduction in activity levels at 10 minutes.

As shown in Figure 4.13, the activity in the >6.3 mm diameter fraction decreases from 0.5 pCi ^{241}Am/g to less than 0.1 pCi ^{241}Am/g following 5 minutes of autogenous grinding. This level increased to 0.15 pCi ^{241}Am/g following an additional 5 minutes of grinding. This increase is most likely due to sampling and/or counting statistics. The actual removal of activity from the sample can be observed by examining the activity measurements on the <6.3 mm diameter material which was removed from the drum following each grinding stage. The <6.3 mm diameter soil mass recovered from each stage of grinding were 46.9 grams (1.9% of total mass), 17.1 grams (0.72%), and 8.8 grams (0.37%) for the 2, 3, and 5 minute retention times, respectively. Although the activity concentration (pCi ^{241}Am/g) increases with time, the mass of <6.3 mm diameter material decreases, resulting in a decrease in total activity removed with each time increment to 5 minutes. Overall, autogenous grinding was successful in removing surface deposited plutonium from >6.3 mm gravel to reducethe plutonium level to less than the 0.9 pCi $^{239+240}$Pu/g criteria.

4.1.3.2 Attrition Scrubber Testing Results and Discussion

The results of the first tests are shown in Figure 4.14. The activity levels show a decrease in activity level for the first 10 minutes of the scrubbing, similar to the autogenous grinding test. Activity is transferred from the larger diameter soil fractions to the <0.15 mm fraction as it is scrubbed from the surface.

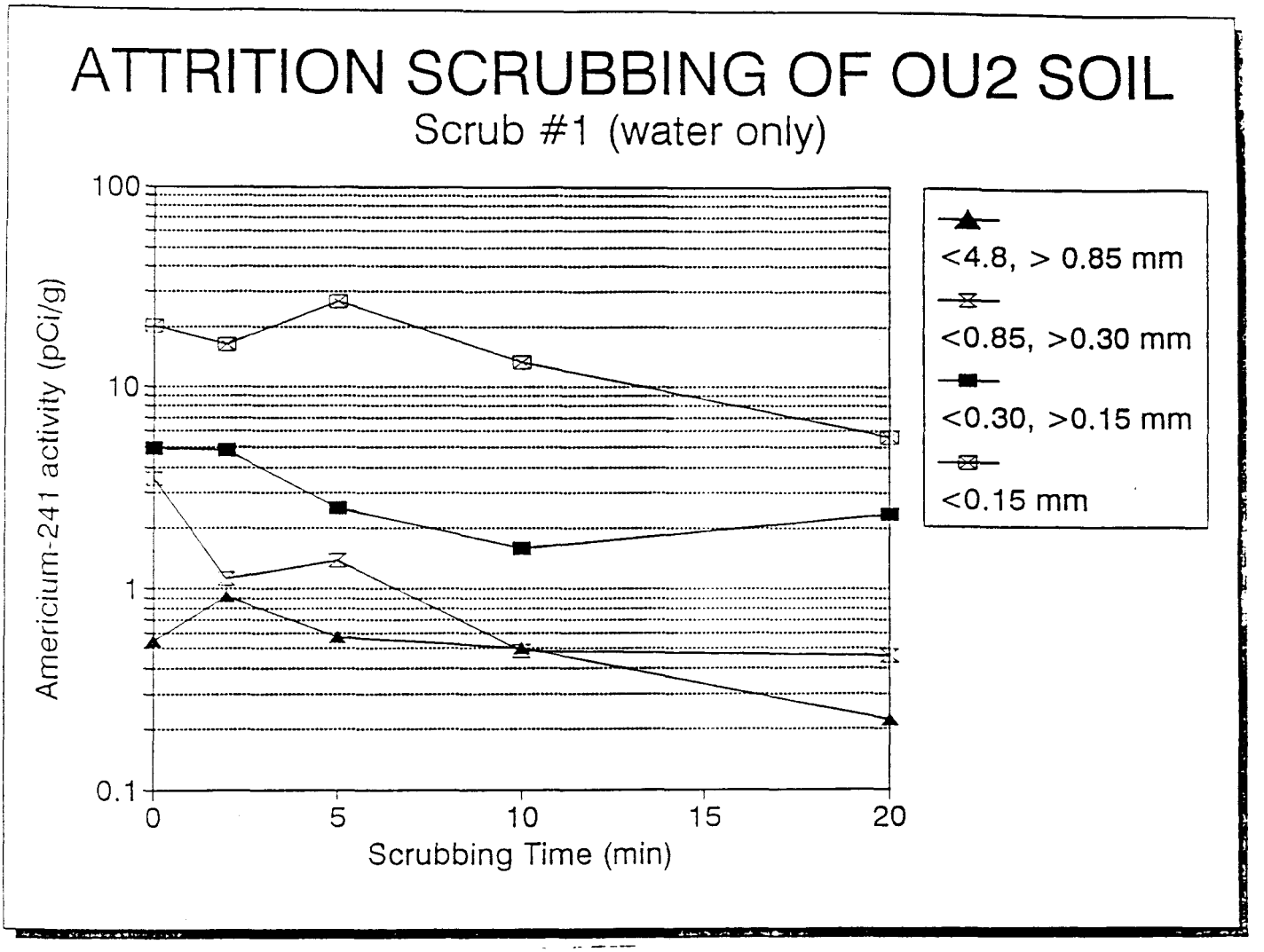
As the scrubbing time is increased, the activity/mass level for the >0.15 mm fractions decreases as activity is again transferred to the <0.15 mm fraction. A longer scrubbing time was thus indicated and was chosen for the bench scale attrition scrubber for reducing the activity level to the lowest possible level while recovering as much clean soil as



Autogenous Grinding Test Results
Starting Activity = 0.5 pCi^{241}Am/g

SCRUB TIME (MIN)	<31.8 mm >6.3 mm		<6.3 mm SOIL	
	^{241}Am (pCi/g)	Mass (g)	^{241}Am (pCi/g)	Mass (Grams)
0	0.5		0.5	0
2	0.54	2363	4.85	46.9
5	0.09	2363	4.84	17.1
10	0.15	2369	10.9	8.8

Figure 4.13



Attrition Scrubbing Test #1 Results
Starting Activity = 2.66 pCi ^{241}Am/g

	<4.8 mm >0.85 mm SOIL		<0.85 mm >0.30 mm SOIL		<0.30 mm >0.15 mm SOIL		<0.15 mm SOIL	
SCRUB TIME (MIN)	^{241}Am (pCi/g)	MASS (g)	^{241}Am (pCi/g)	MASS (g)	^{241}Am (pCi/g)	MASS (g)	^{241}Am (pCi/g)	MASS (g)
0	0.55	——	3.51	——	4.92	——	20.18	0
2	0.92	403.5	1.12	178.3	4.84	127.5	16.3	47.4
5	0.571	329.7	1.4	207.9	2.51	146.7	26.7	21.3
10	0.502	323.0	0.487	196.5	1.59	150.6	13.3	11.2
20	0.223	307.2	0.462	199.5	2.348	135.2	5.58	25.4

Figure 4.14

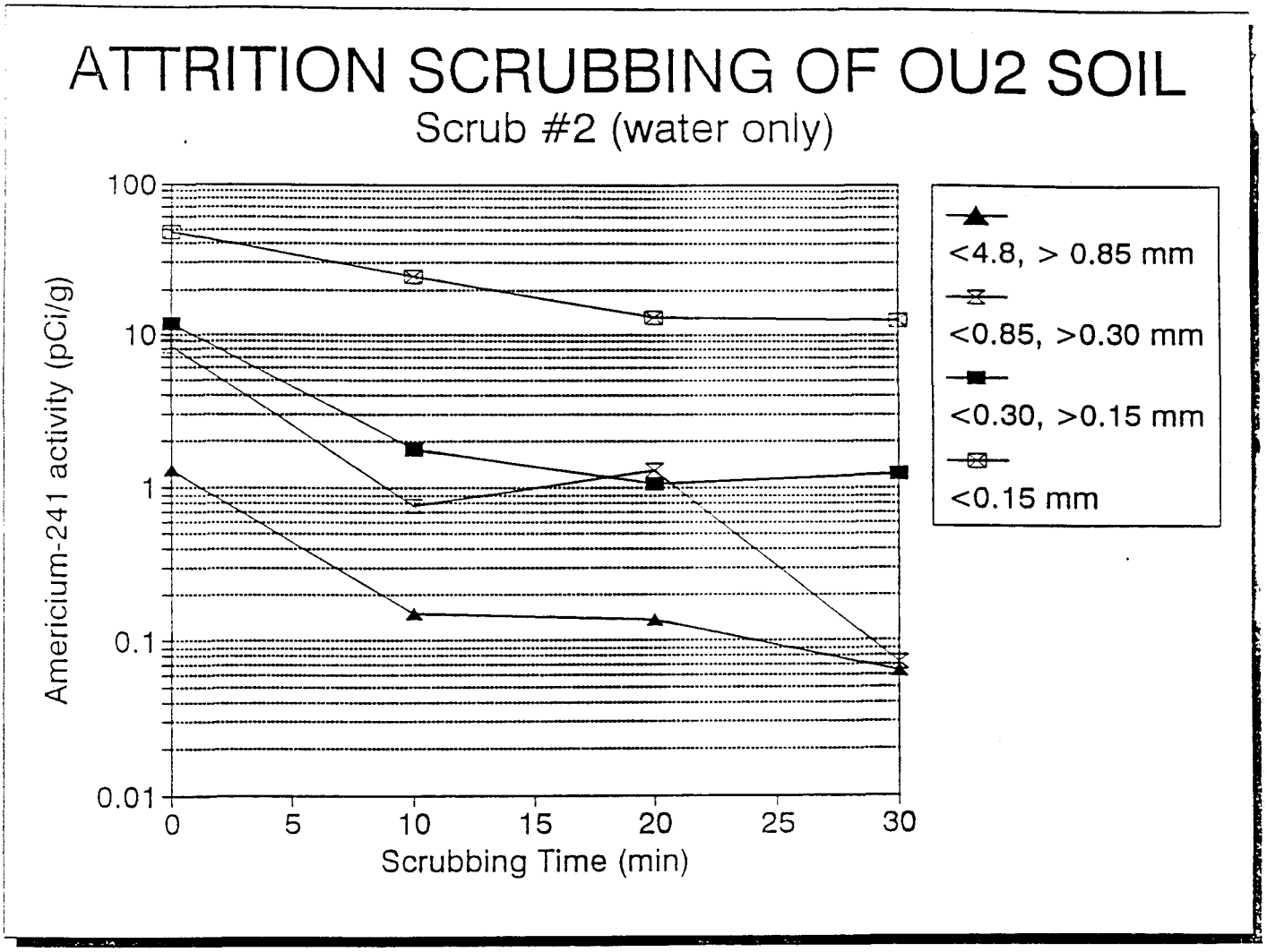
possible. The decreases in activity/mass levels for the <0.15 mm soil at 10 and 20 minutes probably were the result of lower activity material being removed from beneath the originally higher activity outer surfaces on the >0.15 mm material. This trend is the opposite of that observed for autogenous grinding, and may indicate thinner plutonium bearing surfaces on the smaller diameter gravel and sand, as expected. The fluctuations observed in the data for the >0.85 mm and >0.30 mm samples are believed to result from counting statistics and surface conditions of the sample.

A second attrition scrub test was performed to determine reproducibility of data from the first test and to extend scrubbing times to 30 minutes. The data from this test is presented in Figure 4.15. The activity transfer to the <0.15 mm fraction continued on the second scrub test at 30 minutes. Data trends are compatible from Scrub Test 1 to Scrub Test 2 when comparing scrub times of 10 and 20 minutes and starting activities of sample.

The third attrition scrubbing test used the addition of NaOH to attain a slurry pH of 12.0 to 12.5 as shown in Figure 4.16. The data indicated a rapid transfer of activity to the <0.15 mm fraction followed by approximately level activities for the next 10 minutes of scrubbing. The resulting activity concentrations following 30 minutes of scrubbing were comparable or above the first two scrub tests using only water. The economics and environmental compatibility of using NaOH is questionable and further testing was not carried out.

4.1.3.3 Mineral Jig/Spiral Classifier Results and Discussion

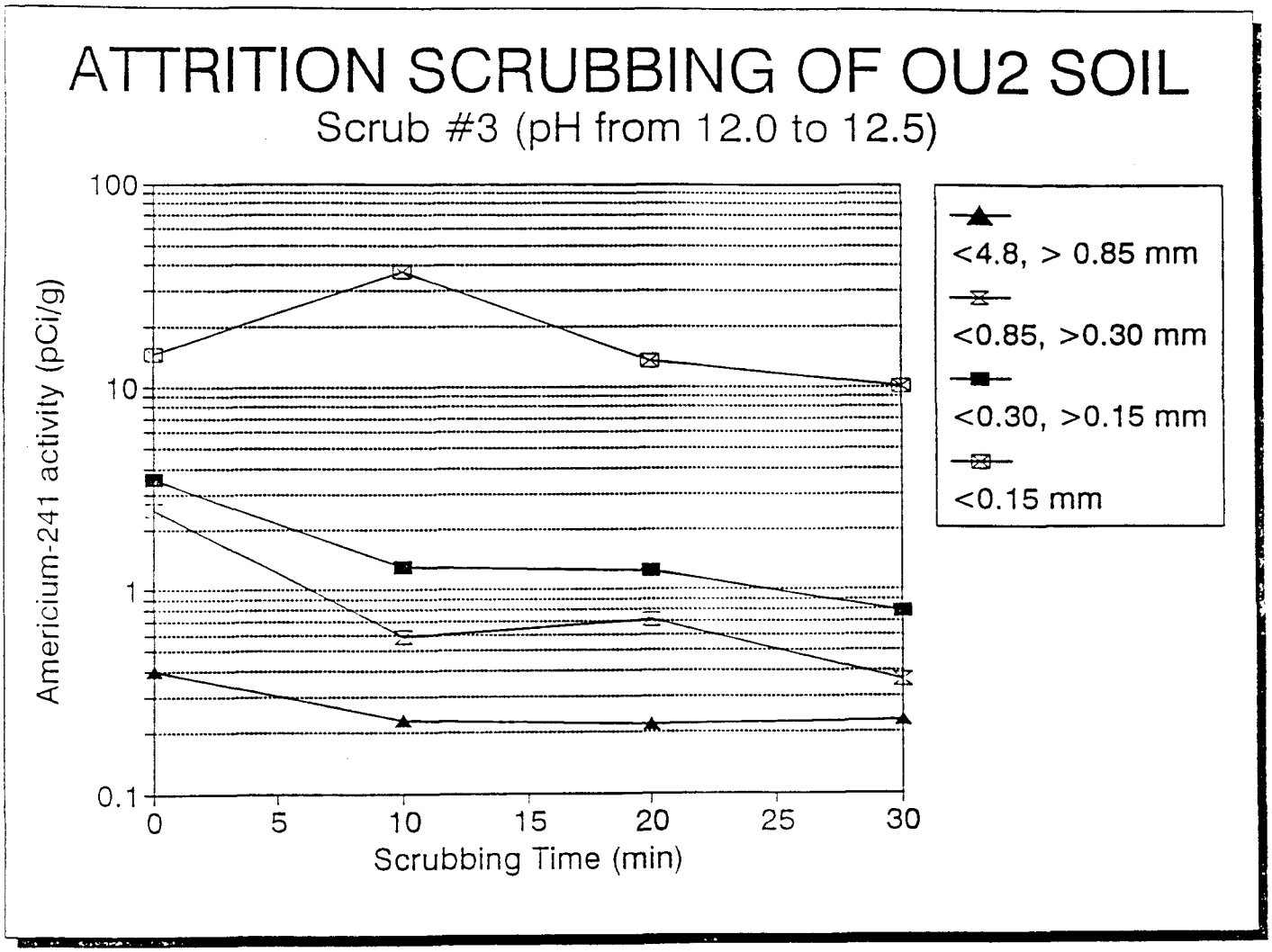
The results of the test are shown in Table 4.8. During the test, five variables were evaluated to determine settings which provided the highest recoveries of plutonium surrogates. These variables were jig stroke length, stroke frequency, hutch 1 flowrate, hutch 2 flowrate, and surrogate particle size. Previous testing and operating characteristics of the mineral jig allowed the tests to be carried out on a limited number of settings. The data indicates recovery of the 50 grams of magnetite used in each test decreases with particle size. The 45 to 75 micron diameter magnetite approaches the minimum particle size which can be recovered using the mineral jig.



Attrition Scrubbing Test #2 Results
Starting Activity = 6.35 pCi ^{241}Am/g

	<4.8 mm >0.85 mm SOIL		<0.85 mm >0.30 mm SOIL		<0.30 mm >0.15 mm SOIL		<0.15 mm SOIL	
SCRUB TIME (MIN)	^{241}Am (pCi/g)	MASS (g)	^{241}Am (pCi/g)	MASS (g)	^{241}Am (pCi/g)	MASS (g)	^{241}Am (pCi/g)	MASS (g)
0	1.31		8.38		11.75		48.16	0
10	0.15	565.6	0.77	262.7	1.76	183.7	24.7	126.4
20	0.137	532.4	1.3	267.1	1.06	170.7	13.0	27.5
30	0.064	533.0	0.073	261.5	1.23	157.0	12.5	15.6

Figure 4.15



Attrition Scrubbing Test #3 Results
Starting Activity = 1.92 pCi ^{241}Am/g

	<4.8 mm >0.85 mm SOIL		<0.85 mm >0.30 mm SOIL		<0.30 mm >0.15 mm SOIL		<0.15 mm SOIL	
SCRUB TIME (MIN)	^{241}Am (pCi/g)	MASS (g)	^{241}Am (pCi/g)	MASS (g)	^{241}Am (pCi/g)	MASS (g)	^{241}Am (pCi/g)	MASS (g)
0	0.40	——	2.53	——	3.55	——	14.56	0
10	0.23	488.9	0.59	265.2	1.3	170.2	37.4	59.43
20	0.22	482.5	0.71	280.3	1.25	144.8	13.45	13.1
30	0.23	480.7	0.36	269.9	0.78	144.8	10.1	11.9

Figure 4.16

Table 4.8 Removal of Plutonium Surrogate from Soil

Surrogate Run Number	Stroke Length (cm)	Stroke Frequency (cycles/min)	Hutch 1 Flow (L/min)	Hutch 2 Flow (L/min)	Magnetite Particle Size (micron)	Magnetite Recovery (%)
3	0.95	100	9.5	9.5	150-300	71.0
4	0.95	150	9.5	9.5	150-300	84.2
5	0.95	150	11.4	11.4	150-300	82.8
6	0.95	150	13.2	13.2	150-300	89.6
7	0.635	150	11.4	11.4	75-150	67.0
8	0.635	200	11.4	11.4	75-150	66.6
9	0.635	150	11.4	11.4	75-150	78.2
10	0.635	150	11.4	11.4	45-75	24.8
11	0.635	150	13.2	13.2	75-150	78.0
12	0.635	150	7.6	7.6	45-75	21.8

Since any plutonium particles recovered from soil are expected to be small, stroke lengths and frequencies were varied within previously successful settings to determine optimum values for small diameter plutonium. A stroke frequency of 150 cycles per minute provided the best recoveries for the 75-150 micron and 150-300 micron diameter particles. Shorter stroke lengths are typically used for recovery of smaller particles.

The best hutch water flows were found to be 13.2 liters per minute for the 150-300 micron diameter particles and 11.4 liters per minute for the 45-75 micron and 75-150 micron diameter particles. Additional information on this test is provided in Appendix A.

4.1.4 Data Analysis of Phase 2 Treatability Study Data

The integrated results for Phase 2 test runs are depicted in Figure 4.17. The results for individual process equipment and output streams are discussed in the following subsections. Testing sequence should be referenced to Figure 4.17.

4.1.4.1 Dry Screening Test Results and Discussion

Four Phase 2, dry screening tests were performed which utilized about 90.8 kg of soil for each test. Figures 4.18 and 4.19 illustrate the screening process along with soil mass and resulting activities for each soil stream. Test data and mass/activity balances are shown in Table 4.9. The organic stream specified in the figure contained naturally occurring undecomposed organics (grass and roots) which were manually removed from the >31.8 mm opening screen. These organics

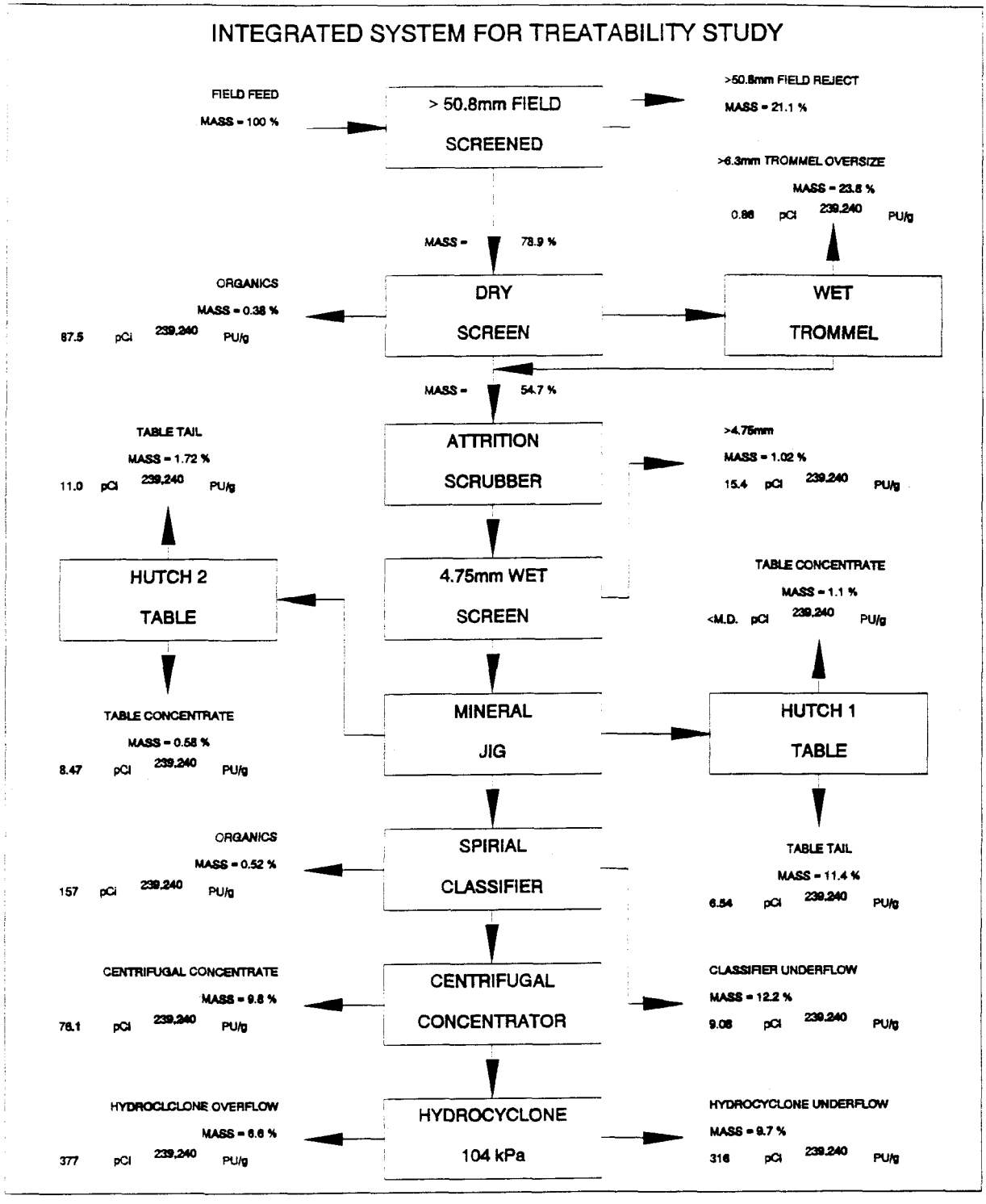


Figure 4.17 Integrated Results of Phase Tests

Process Stream Name	Percent Mass	Activity (pCi $^{239, 240}$Pu/g)
>51 mm/Field Screened	21.1%	------
>32mm/Dry Screened Organics	0.38	87.5
>6.3mm/Trommel Oversize	23.8	0.86
>4.8mm/Wet Screened	1.02	15.4
Hutch 1, Table Tails	11.4	6.54
Hutch 1, Table Concentrate	1.1	0.0
Hutch 2, Table Tails	1.72	11.0
Hutch 2, Table Concentrate	0.58	8.47
Spiral Classifier, Screened Organics	0.52	157.0
Spiral Classifier Underflow	12.2	9.08
Centrifugal Concentrate	9.8	76.1
Hydrocyclone Overflow	6.6	377.0
Hydrocyclone Underflow	9.7	316.0

Integrated Results of Phase 2 Tests.

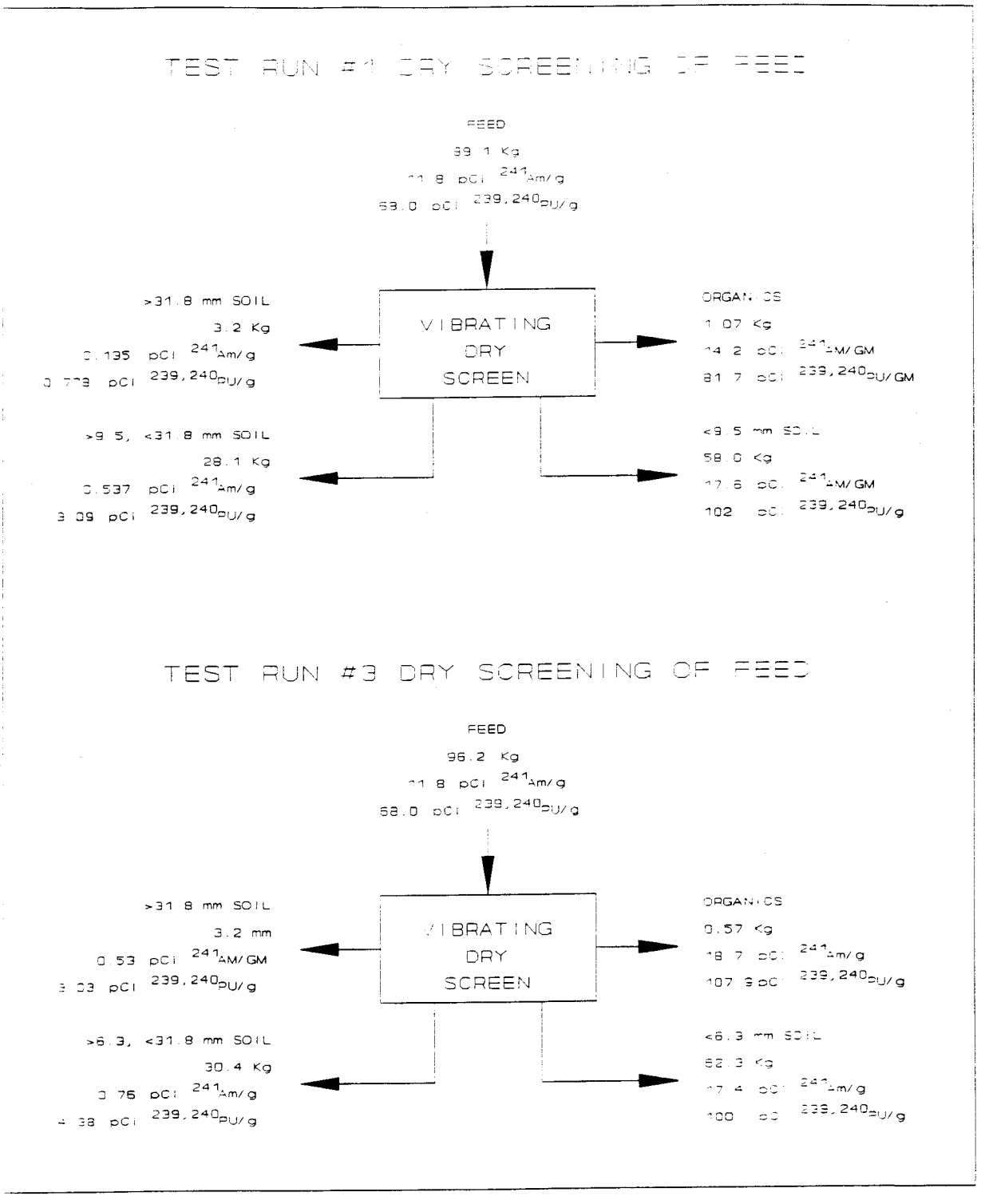


Figure 4.18 Results of Dry Screening for Runs 1 and 3

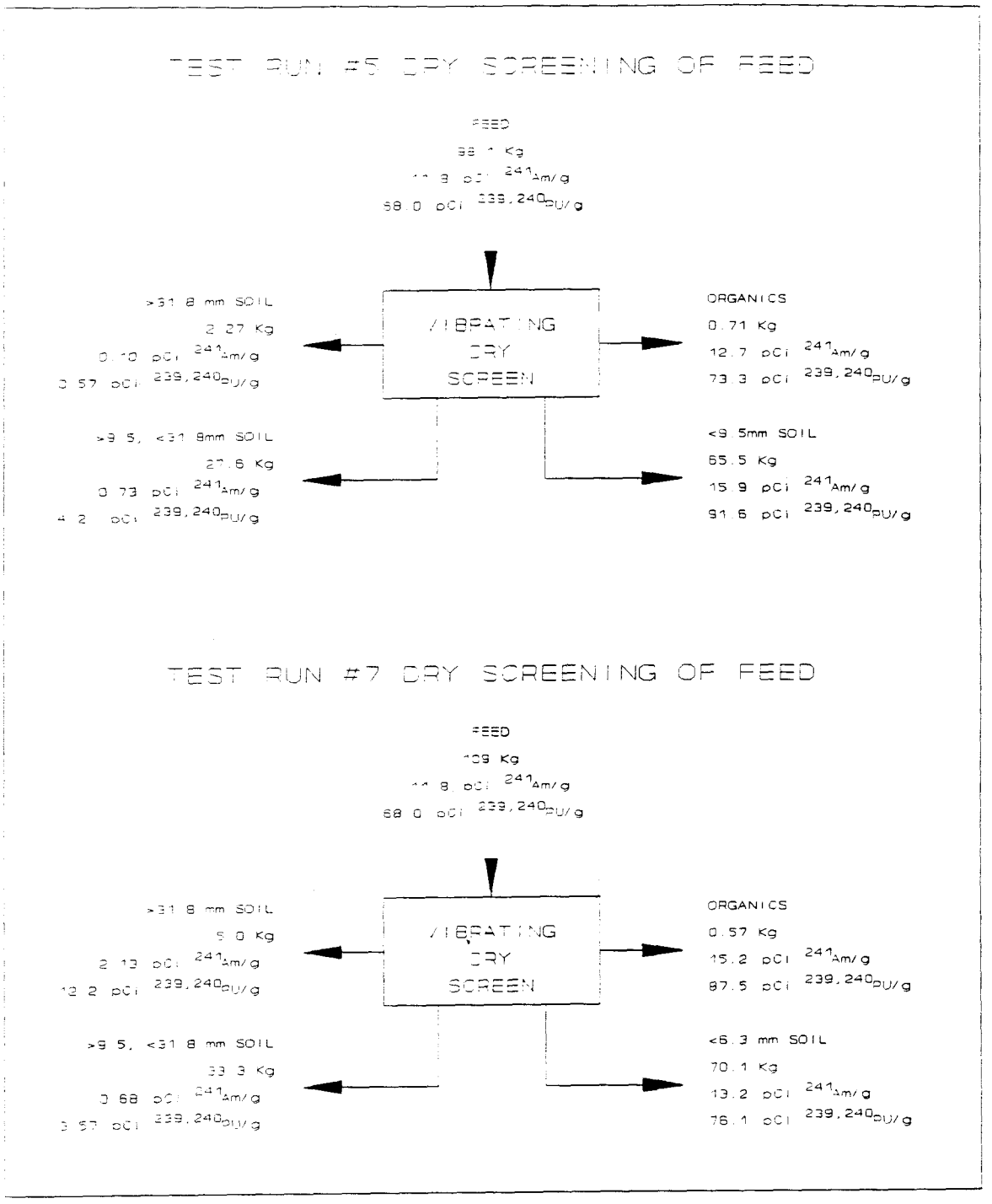


Figure 4.19 Results of Dry Screening for Runs 5 and 7

Table 4.9 Results of Dry Screening Tests

RUN NO.	PROCESS STREAM	PROCESS STREAM MASS (Kg)	% MASS	PROCESS STREAM (pCi ^{241}Am/g)	^{241}Am ACTIVITY (pCi)	^{239,240}Pu ACTIVITY (pCi/g)	% TOTAL ACTIVITY
1	>31.8mm Organics	1.07	1.2	14.2	15,100	81.7	1.4
	>31.8mm Inorganic	3.2	3.6	0.14	429	0.78	<0.1
	>9.5mm Gravel	28.1	31.5	0.54	15,100	3.1	1.4
	<9.5mm Soil	58.0	65.1	17.6	1,020,000	102.0	97.0
	Screen Cleanout	0.82	1.0	0.54	430	3.1	<0.1
	TOTAL	91.2	102.4	———	1,050,000	———	99.8
1	Feed	89.1	100.0	11.8	1,051,400	69.2	100.0
3	>31.8mm Organics	0.57	0.6	18.7	10,700	108.0	0.9
	>31.8mm Inorganic	3.2	3.3	0.53	1,680	3.0	0.1
	>6.3mm Gravel	30.4	31.6	0.76	23,000	4.4	2.0
	<¼ Soil	62.3	64.8	17.4	1,080,000	100.0	95.1
	Screen Cleanout	4.4	4.6	17.4	77,100	98.7	6.7
	TOTAL	100.9	104.9	———	1,192,000	———	104.8
3	Feed	96.2	100.0	11.8	1,135,200	69.2	100.0
5	>31.8mm Organics	0.71	0.7	12.7	8,980	73.3	0.8
	>31.8mm Inorganic	2.27	2.3	0.1	218	0.57	<0.1
	>6.3mm Gravel	27.6	28.1	0.73	20,200	4.2	1.7
	<6.3mm Soil	65.5	66.8	15.9	1,038,000	91.6	89.7
	Screen Cleanout	———	———	———	———	———	———
	TOTAL	96.1	97.9	———	1,067,000	———	92.2
5	Feed	98.1	100.0	11.8	1,157,600	69.2	100.0
7	>31.8mm Organics	0.57	0.5	15.2	8,690	87.5	0.7
	>31.8mm Inorganic	5.0	4.6	2.1	10,600	12.2	0.8
	>6.3mm Gravel	33.3	30.6	0.68	22,600	3.6	1.8
	<6.3mm Soil	70.1	64.3	12.9	904,300	80.1	70.3
	Screen Cleanout	7.81	7.2	13.9	108,500	80.1	8.4
	TOTAL	116.8	107.2	———	1,054,700	———	82.0
7	Feed	109.0	100.0	11.8	1,286,200	69.2	100.0

contained substantially elevated levels of americium (12.7 to 18.7 pCi ^{241}Am/g).

The >31.8 mm and <31.8 mm, >9.5 mm or >6.3 mm diameter gravel was combined and processed through a trommel screen as described in Section 4.1.4.2. The minus 6.3 or minus 9.5 mm material was used as feed material for the mineral jig test described in Section 4.1.4.3.

The screening process isolated between 31% and 35% of the soil mass as gravels greater than 9.5 or 6.3 mm diameter. The soil passing the lower screen (<9.5 or <6.3 mm diameter) typically contained greater than 90% of the soil activity. As a result of the screening process, the soil passing through both screens increased in plutonium activity from 76 pCi/g whole soil (<5.1 cm) to as high as 102 pCi/g.

The gravels retained on the screens had a residual plutonium activity up to 4.38 pCi/g. The values presented in the figures represent individual analysis of samples or the average of two or more samples when obtained. The test runs which had >100% recovery on these tests were the result of soil materials which were caught in the screen and recovered during the following test run. A detailed spreadsheet containing individual sample analysis is provided in Appendix A. Photographs of materials resulting from this screening test are also found in Appendix A.

4.1.4.2 Trommel Test Results and Discussion

The mass and activity distributions from the trommel test are shown in Figures 4.20 and 4.21. The mass and activity balances are shown in Table 4.10.

Activity levels on the soil fed to the trommel were between 2.24 and 7.26 pCi$^{239+240}$ Pu/g. Activity levels in the >6.3 mm diameter gravels leaving the trommel was reduced to between 0.86 and 4.73 pCi $^{239+240}$ Pu/g. The activity removed from the surfaces of gravel in the trommel can be observed by examining the mass and activity levels of the <6.3 mm diameter fraction. The activity in the <6.3 mm diameter material was substantially elevated compared with the trommel feed and was of a small mass percentage.

To provide a mass and activity balance for the tests, the residual material remaining in the trommel scrubber and screen sections was removed at test completion and analyzed. These values are shown in Figures 4.20 and 4.21 and in Table 4.10. If the test would have been extended, most of these materials would find their way to the >6.3 mm diameter-material discharge chute on the trommel. Therefore, the mass and activity found in these heels has been added to the >6.3 mm diameter fraction for the overall performance evaluation of the integrated system described in Section 4.1.5.

As indicated in Section 4.1.2.5, the method of analysis of samples was gamma spectroscopy. The values presented in the Figures 4.20 and

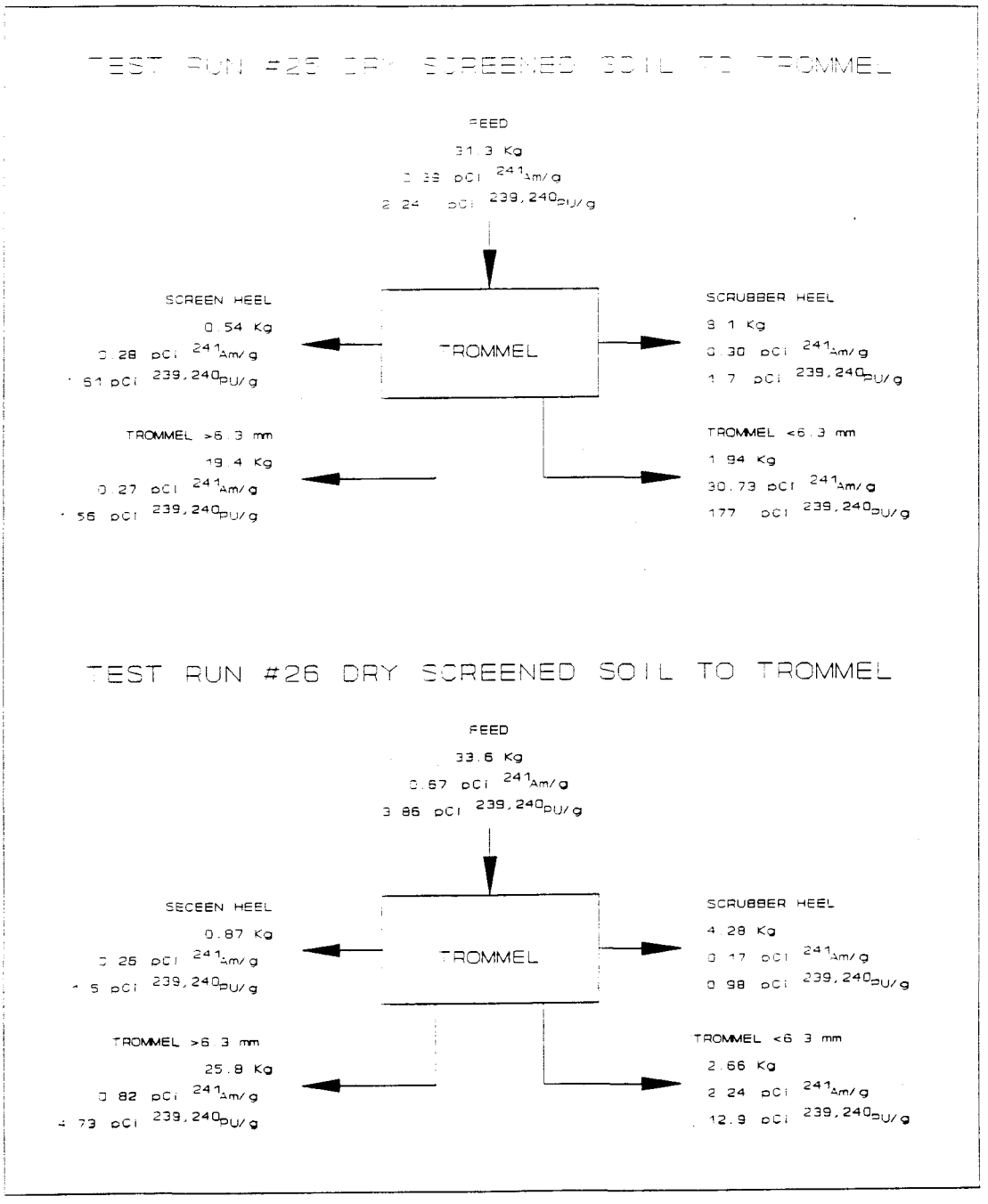


Figure 4.20 Results of Trommel Scrubbing/Screening for Runs 25 and 26

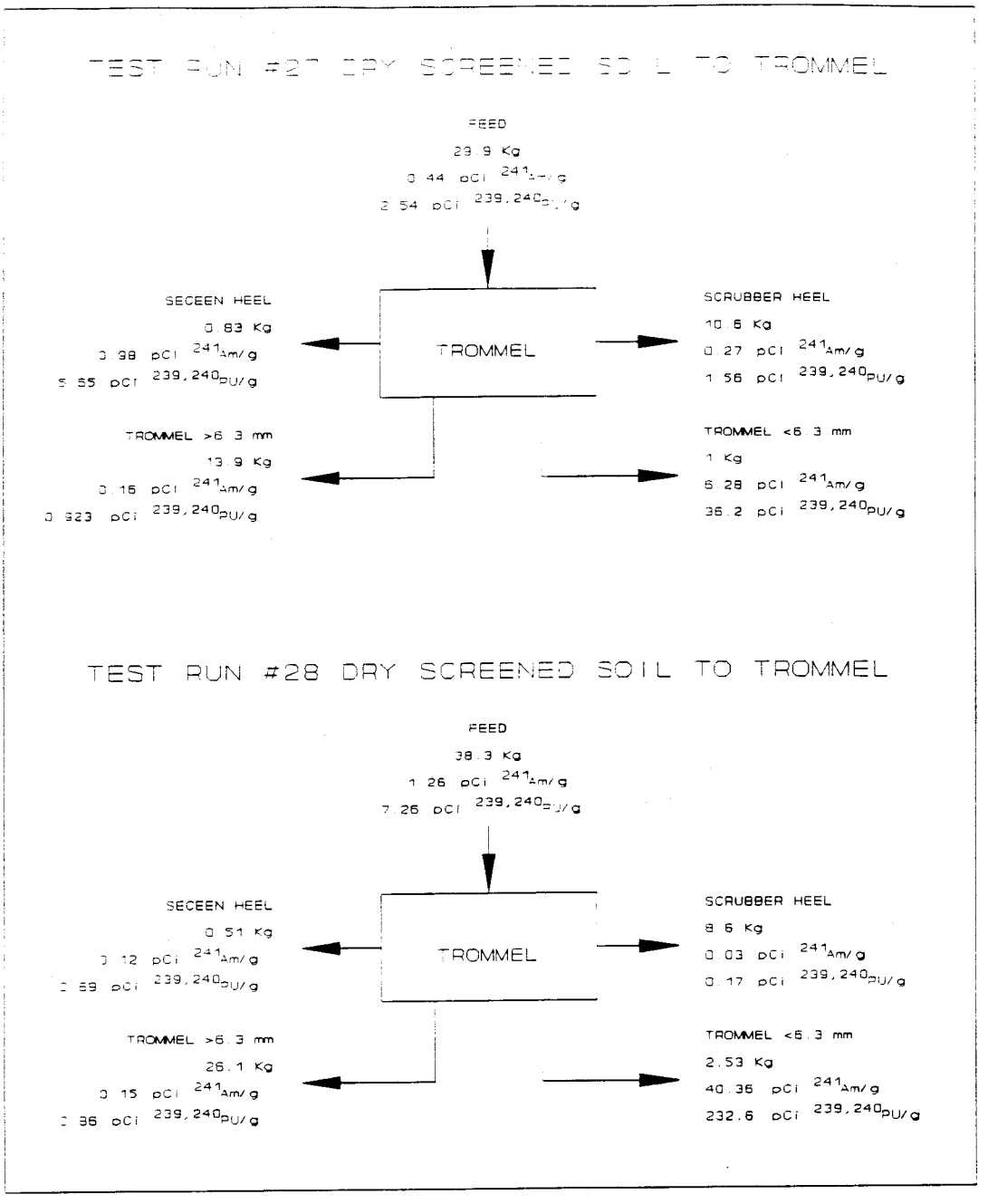


Figure 4.21 Results of Trommel Scrubbing/Screening for Runs 27 and 28

Table 4.10 Results of Trommel Scrubbing/Screening Tests

RUN NO.	PROCESS STREAM	PROCESS STREAM MASS (Kg)	% MASS	PROCESS STREAM (pCi ^{241} Am/g)	^{241}Am ACTIVITY (pCi)	^{239,240}Pu ACTIVITY (pCi/g)	% TOTAL ACTIVITY
25	>6.3mm Oversize	19.4	62.0	0.27	5,200	1.6	42.6
	<6.3mm Undersize	2.0	6.4	30.7	59,600	177.0	488.5
	Screen Heel	0.54	1.7	0.28	151	1.6	1.2
	Scrubber Heel	8.1	25.9	0.30	2,380	1.7	19.5
	TOTAL	30.1	96.0	———	67,300	———	551.6
25	Feed	31.3	100.0	0.39	12,200	2.2	100.0
26	>6.3mm Oversize	25.8	76.8	0.82	21,200	4.7	95.1
	<6.3mm Undersize	2.7	8.0	2.24	6,000	12.9	26.9
	Screen Heel	0.86	2.6	0.26	220	1.5	1.0
	Scrubber Heel	4.3	12.8	0.17	720	0.98	3.2
	TOTAL	33.6	100.0	———	28,140	———	126.2
26	Feed	33.6	100.0	0.67	22,300	3.9	100.0
27	>6.3mm Oversize	13.9	46.5	0.16	2,260	0.92	17.0
	<6.3mm Undersize	1.0	3.3	6.28	6,230	36.2	46.8
	Screen Heel	0.83	2.8	0.98	810	5.7	6.1
	Scrubber Heel	10.6	35.4	0.27	2,820	1.6	21.2
	TOTAL	26.3	88.0	———	12,120	———	91.1
27	Feed	29.9	100.0	0.44	13,300	2.5	100.0
28	>6.3mm Oversize	26.1	68.1	0.15	3,900	0.86	8.1
	<6.3mm Undersize	2.5	6.5	40.4	102,000	233.0	211.6
	Screen Heel	0.5	1.3	0.12	61	0.69	0.1
	Scrubber Heel	8.6	22.4	0.03	250	0.17	0.5
	TOTAL	37.7	98.4	———	106,211	———	220.4
28	Feed	38.3	100.0	1.26	48,200	7.3	100.0

4.21 may have some contribution of activity from naturally occurring radioactive materials (NORM) which overlap the 60 keV americium peak. The radiochemical analysis of these fractions provided the most precise measurement of residual plutonium levels from the >6.3 mm diameter fraction.

Feed rates for the trommel tests were initially determined from the Phase 1 tumbling test. These tests indicated a contact time of 5 minutes was sufficient to scrub off most of the plutonium. Test run #27 used a 2 minute residence time with a feed rate of 330 kg per hour. Test runs 25 and 28 used a feed rate of 90.8 kg per hour which increased residence time to around 5 minutes. Test run #26 had a feed rate of 49.5 kg per hour which resulted in a residence time in the scrubber of around 10 minutes. The data does not show any decrease in residual activity as scrubbing time is increased beyond the 2 minute residence time.

4.1.4.3 Attrition Scrubbing, Mineral Jig, Spiral Classifier, and Thickener Test Results and Discussion

Figures 4.22 through 4.25 contain mass and activity distributions for the mineral jig tests. Mass and activity balances are shown in Table 4.11. The mineral jig tests are designated by test runs 2, 4, 6, and 8. Each test run used the material passing both dry screens, as explained in Section 4.1.4.1. The total mass and activity for the tests are based on the mass and activity measurements obtained on the <9.5 mm or <6.3 mm diameter material from the dry screening test. As can be seen from the data in the figures, the majority of the activity stayed with the fine material and was found in the thickener. The activity found in the >4.8 mm product, mineral jig hutches and spiral classifier underflow ranged between 4.5 and 6.4% of the test feed activity. The organic fraction captured by screening the classifier overflow contained between 1.2 and 1.8% of the feed activity. The remainder of the activity was found in the thickener. It should be noted that the classifier cleanout represents a heel which is permanently located in the classifier pool.

For the purposes of an activity balance the activity located in this heel would find its way to the thickener if the test were extended. For purposes of the mass balance the mass located in the heel would eventually be elevated up the spiral classifier incline and be discharged as classifier underflow. This logic follows operating characteristics of the spiral classifier which is used to separate soil particles by size. As can be observed in the data, the classifier underflow typically has an activity concentration of 9 to 19 $pCi^{239+240}$ Pu/g while the classifier overflow collected in the thickener has an activity concentration of 194 to 228 $pCi^{239+240}$ Pu/g. This fraction was further processed and produced a centrifugal concentrate stream at 76.1 $pCi^{239,240}$Pu/g, and hydrocyclone underflow and overflow splits at 318 and 377 $pCi^{239,240}$Pu/g (Table 21. and Figure 4.17).

The mass of soil collected from the >4.8 mm opening diameter screen and the spiral classifier underflow was considered to be a clean stream which was free of liberated contaminants. The >4.8 mm opening

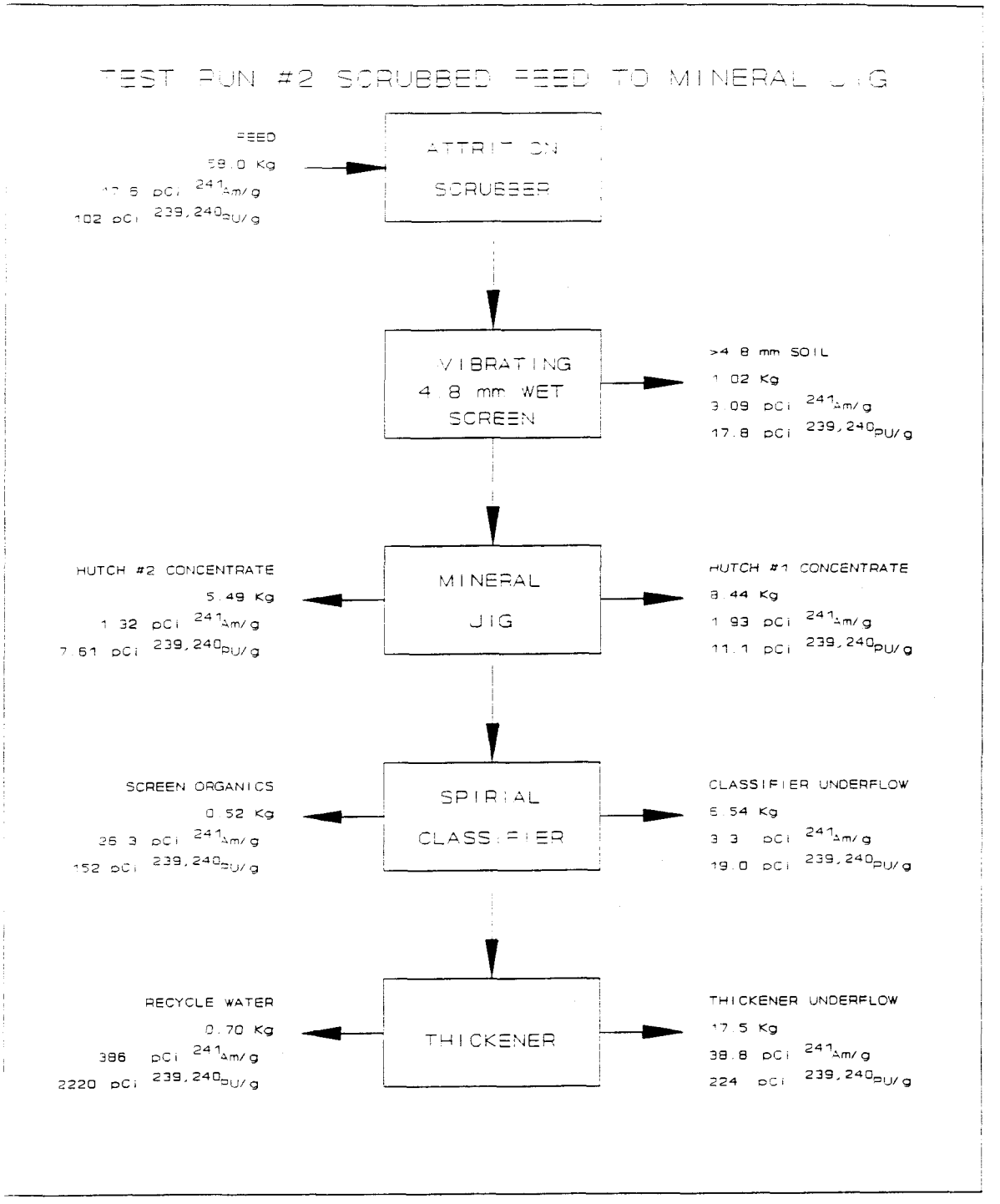


Figure 4.22 Results of Mineral Jig Test Run 2

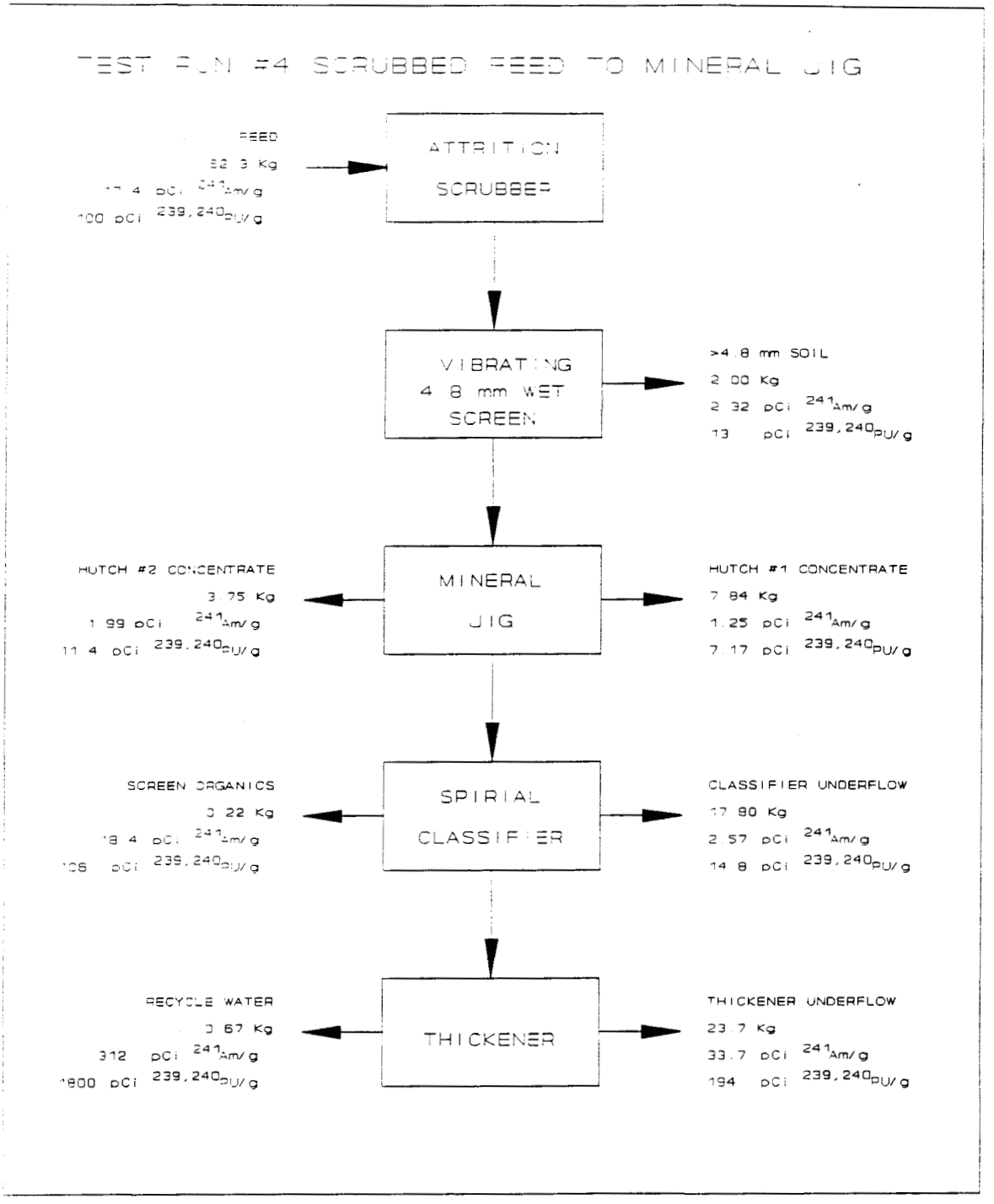


Figure 4.23 Results of Mineral Jig Test Run 4

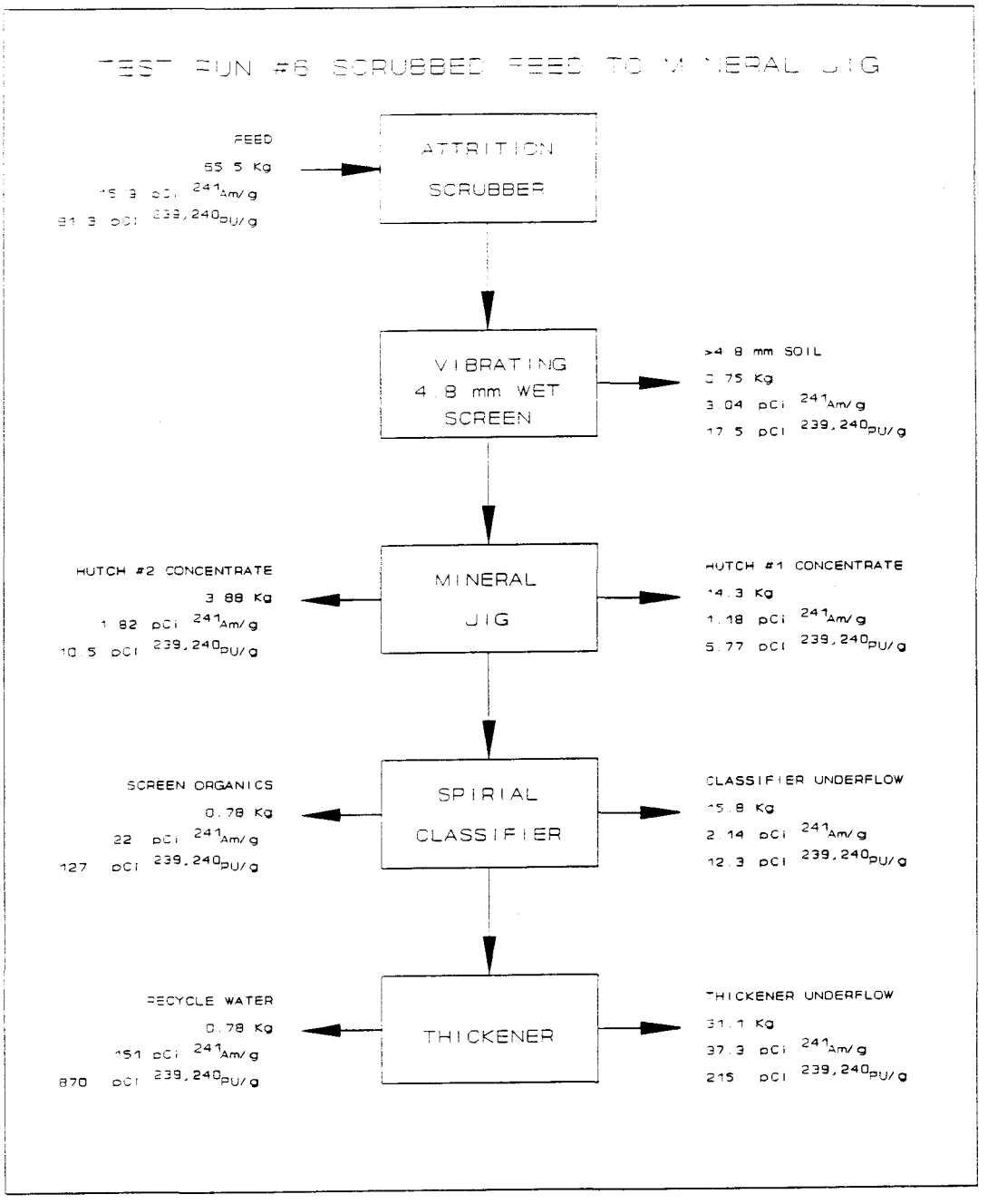


Figure 4.24 Results of Mineral Jig Test Run 6

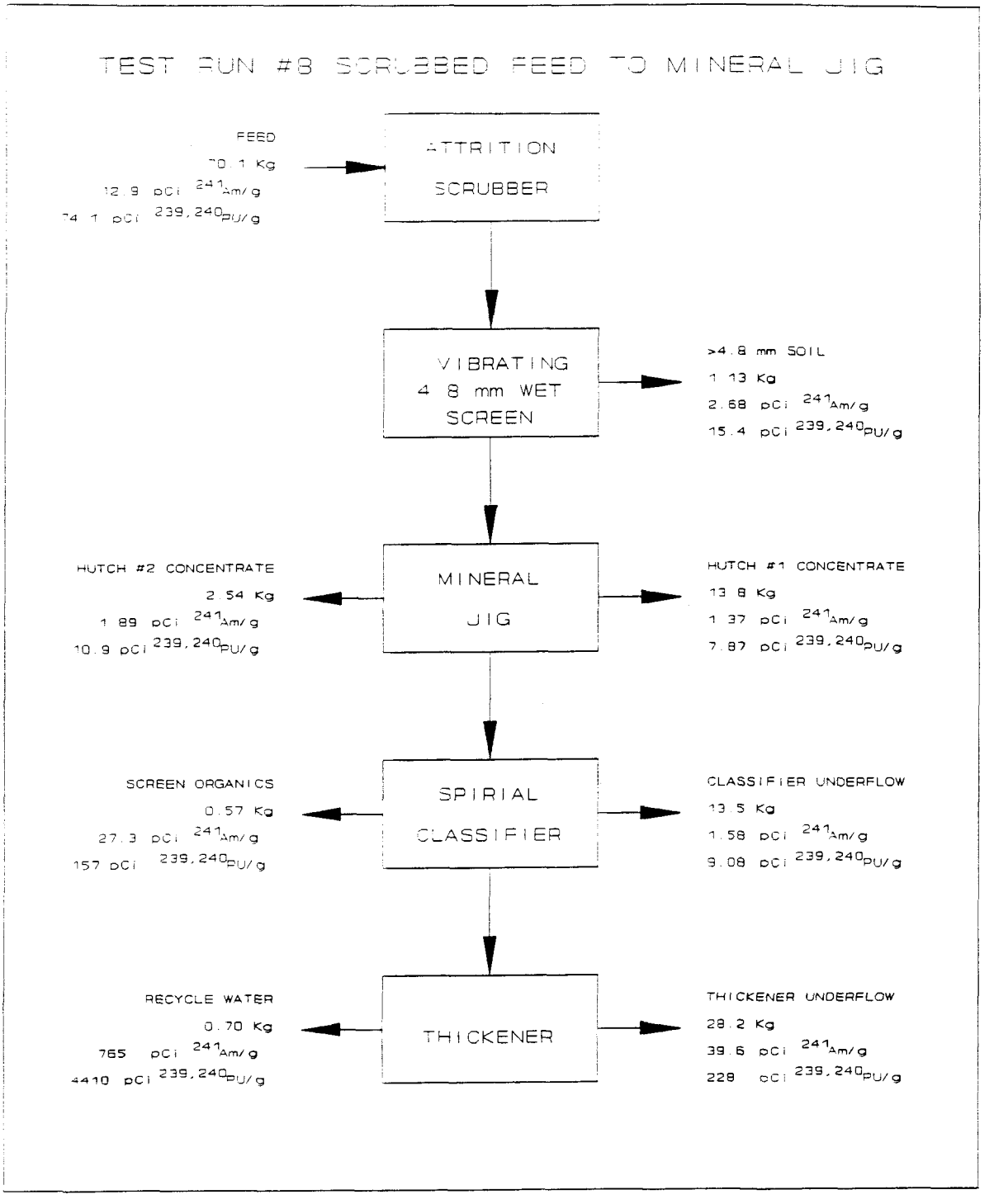


Figure 4.25 Results of Mineral Jig Test Run 8

Table 4.11 Results of Scrubbing, Mineral Jig, and Classification Tests

RUN NO.	PROCESS STREAM	PROCESS STREAM MASS (Kg)	% MASS	PROCESS STREAM (pCi ^{241}Am/g)	^{241}Am ACTIVITY (pCi/g)	^{239,240}Pu ACTIVITY (pCi/g)	% TOTAL ACTIVITY
2	>4.8mm Soil	1.0	1.7	3.09	3080	17.8	0.30
	Hutch 1 Cons	8.4	14.5	1.93	16300	11.1	1.60
	Hutch 2 Cons	5.5	9.5	1.32	7250	7.61	0.71
	Screen Organics	0.5	0.9	26.3	13100	152.0	1.28
	Classifier Underflow	6.5	11.2	3.3	21600	19.0	2.12
	Thickener Underflow	17.5	30.2	38.8	680000	224.0	66.7
	Recycle Water	0.73	1.3	386.0	280000	2220.0	27.4
	TOTAL	40.2	69.4	———	1021330	———	100.1
2	Feed	58.0	100.0	17.6	1020000	102.0	100.0
4	>4.8mm Soil	2.0	3.2	2.32	4600	13.0	0.4
	Hutch 1 Cons	7.8	12.6	1.25	9800	7.17	0.9
	Hutch 2 Cons	3.8	6.0	1.99	7500	11.4	0.7
	Screen Organics	0.2	0.4	18.4	4200	106.0	0.4
	Classifier Underflow	17.8	28.6	2.57	45700	14.8	4.2
	Thickener Underflow	23.7	38.0	33.7	797000	194.0	73.5
	Recycle Water	0.7	1.1	312.0	212000	1800.0	19.5
	TOTAL	56.0	89.9	———	1081000	———	99.6
4	Feed	62.3	100.0	17.4	1085000	100.0	100.0
6	>4.8mm Soil	0.8	1.2	3.04	2300	17.5	0.2
	Hutch 1 Cons	14.3	21.8	1.18	17000	6.8	1.6
	Hutch 2 Cons	3.9	5.9	1.82	7000	10.5	0.7
	Screen Organics	0.8	1.2	22.0	17000	127.0	1.6
	Classifier Underflow	15.8	24.2	2.14	34000	12.3	3.3
	Thickener Underflow	31.1	47.4	37.3	1160000	215.0	111.4
	Recycle Water	0.8	1.2	151.0	117000	870.0	11.2
	TOTAL	67.4	102.9	———	1354000	———	130.1
6	Feed	65.5	100.0	15.9	1041000	91.3	100.0
7	>4.8mm Soil	1.1	1.6	2.68	3000	15.4	0.3
	Hutch 1 Cons	13.8	19.8	1.37	19000	7.9	2.1
	Hutch 2 Cons	2.5	3.6	1.89	4800	10.9	0.5
	Screen Organics	0.6	0.8	27.3	16000	157.0	1.8
	Classifier Underflow	13.5	19.3	1.58	21000	9.1	2.3
	Thickener Underflow	28.2	40.2	39.6	1116000	228.0	123.3
	Recycle Water	0.7	1.0	765	521000	4410.0	57.6
	TOTAL	60.5	86.3	———	1701000	———	187.9
8	Feed	70.1	100.9	12.9	905000	74.1	100.0

diameter and classifier underflow streams contained between 13 and 25% of the soil mass which entered the mineral jig tests. The first test (test run #2) was used to fill the heel of the scrubber and the mass balance reflected a deficit in mass to fill this heel. Neglecting this first test, the mass collected by the two streams in the remaining three tests was 20 to 25% in these two streams. The mineral jig hutch streams collected between 19 and 28% of the feed mass. These streams were further concentrated on a Wilfley Table to further reduce the volume of any contaminants as explained in Section 4.1.4.4. The organic material retained by screening the classifier overflow contained between 0.4 and 1.2% of the feed mass. The remainder of the mass was found in the thickener.

Photographs of the materials found in the test streams and the test equipment are found in Appendix A. A detailed listing of the data collected during the test runs is also found in spreadsheets located in Appendix A.

4.1.4.4 Wilfley Table Test Results and Discussion

The Wilfley Table test were performed as runs #9 through 16. These tests represent tabling of hutches 1 and 2 from runs #2, 4, 6, and 8. The measurements of mass and activity from these runs are shown in Figures 4.26 through 4.29. Mass and activity balances are shown in Table 4.12. Examination of the data in the figures indicates the concentration of the table tails and concentrate are the same activity levels as the classifier underflow. This indicates little, if any, liberated plutonium was present from this sample in the size range for the mineral jig to recover.

Larger particles of plutonium may exist closer to the site of original release and the test results may vary with sample location. Small particles of plutonium or molecular plutonium attached to soil particles will be entrained from the surface and carried further by wind currents as compared to larger particles of plutonium. Larger particles of plutonium will also be retained closer to the release site during water erosion due to their high density. These phenomenon were observed at the Nevada Test Site (Murarik, et al, 1991) and at Johnston Atoll (Wenstrand, 1989).

Photographs of the table tails and concentrates are found in Appendix A.

4.1.4.5 Centrifugal Concentrator and Hydrocyclone Results and Discussion

Centrifugal concentrator and hydrocyclone test results are shown in Figures 4.30 through 4.33. Mass and activity balances are shown in Table 4.13. Test runs 17, 19, 21, and 23 represent centrifugal concentrator tests on thickener underflow from test runs 2, 4, 6, and 8, respectively (<0.075 mm material). Test runs 18, 20, 22, and 24 are hydrocyclone tests on concentrator tails from runs 17, 19, 21, and 23, respectively.

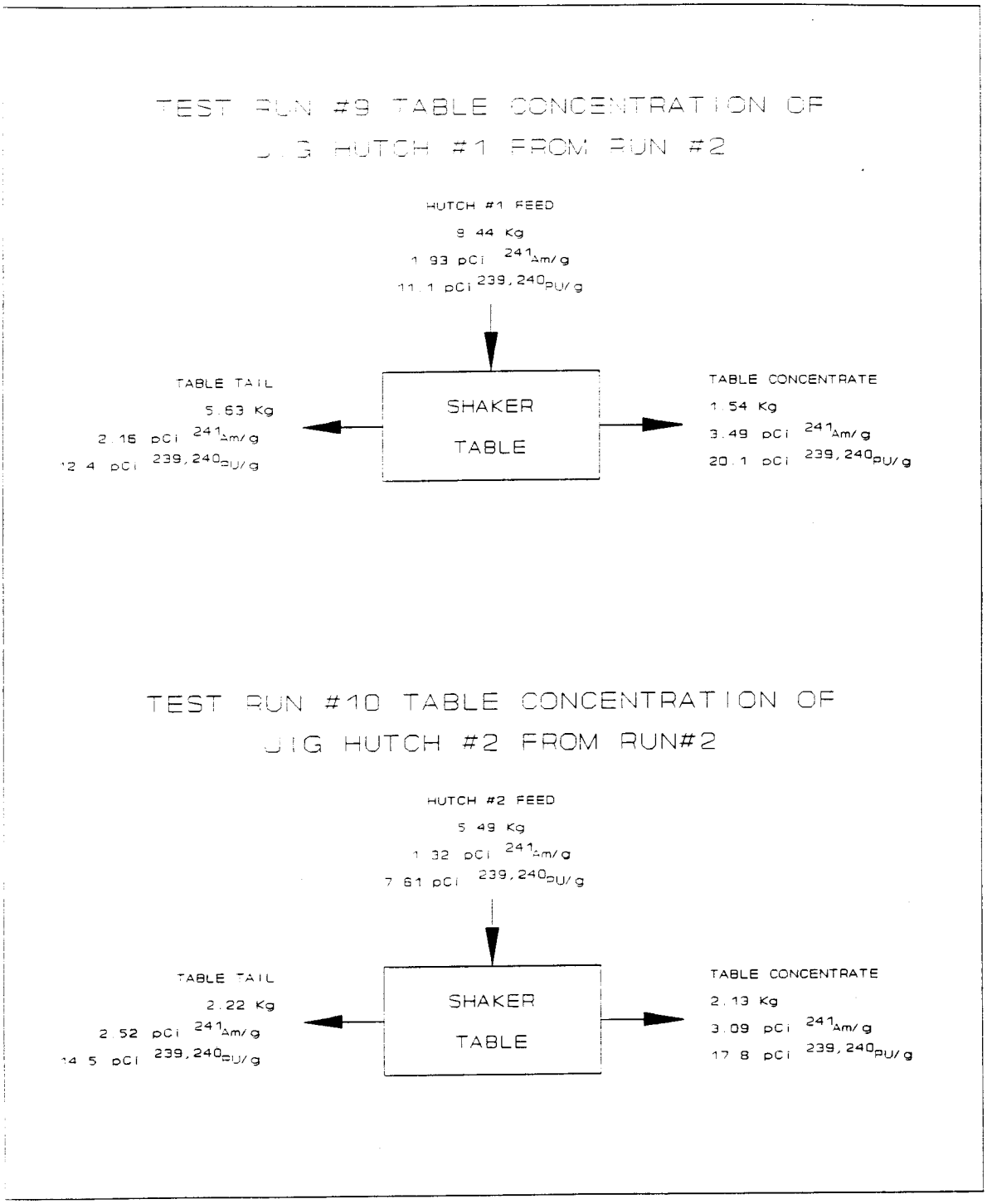


Figure 4.26 Results of Table Concentration of Mineral Jig Hutches from Run 2

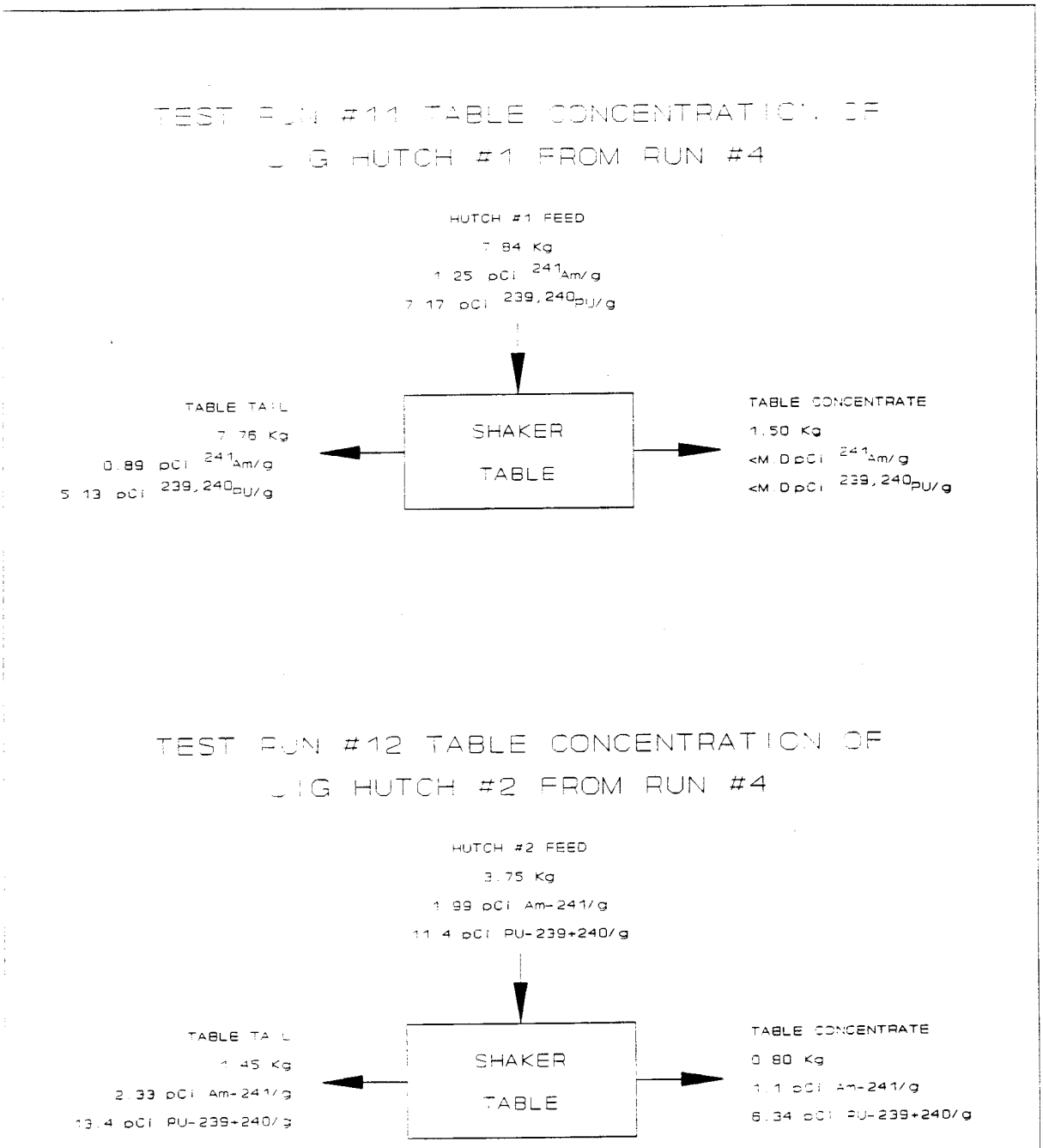


Figure 4.27 Results of Table Concentration of Mineral Jig Hutches from Run 4

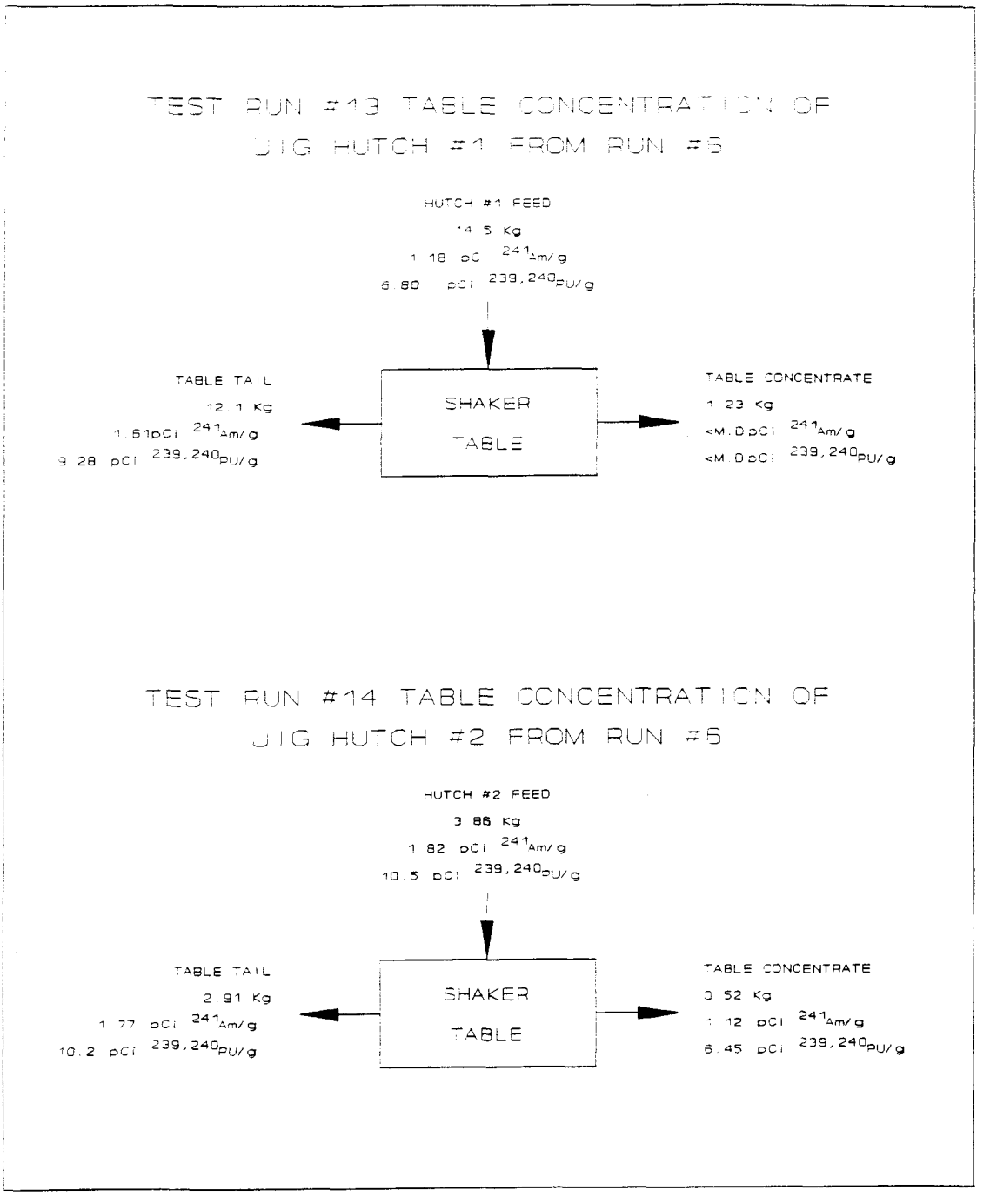


Figure 4.28 Results of Table Concentration of Mineral Jig Hutches from Run 6

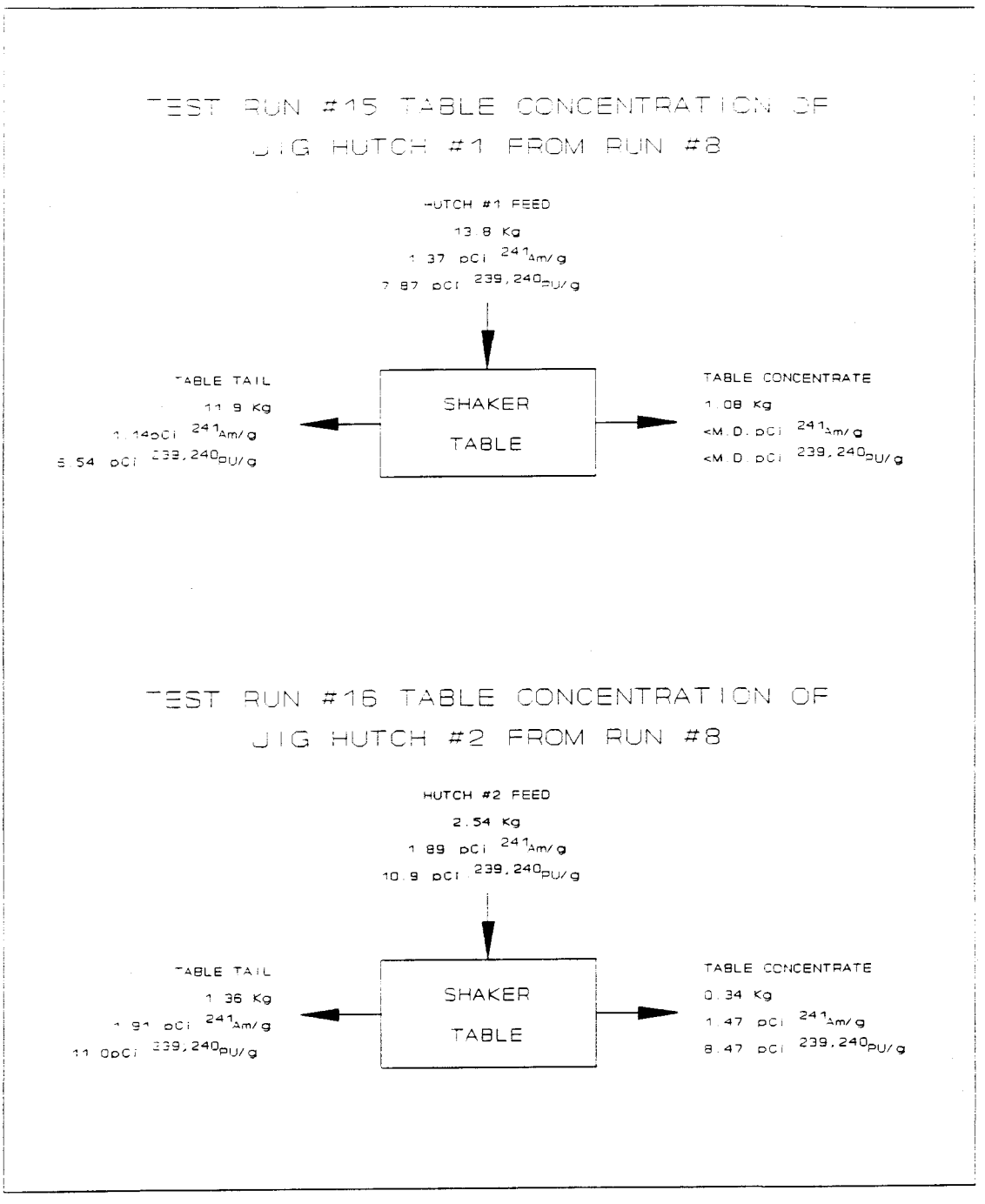


Figure 4.29 Results of Table Concentration of Mineral Jig Hutches from Run 8

Table 4.12 Results of Wilfley Table Tests

RUN NO.	PROCESS STREAM	PROCESS STREAM MASS (Kg)	% MASS	PROCESS STREAM (pCi ^{241}Am/g)	^{241}Am ACTIVITY (pCi)	^{239,240}Pu ACTIVITY (pCi/g)	% TOTAL ACTIVITY
9	Table Tails	5.6	67.0	2.16	12,100	12.4	74.2
	Concentrate	1.5	17.9	3.49	5,370	20.1	33.0
	TOTAL	7.1	84.9	———	17,470	———	107.2
9	Feed	8.4	100.0	1.93	16,300	11.1	100.0
10	Table Tails	2.2	40.5	2.52	5,640	14.5	77.7
	Concentrate	2.1	38.9	3.09	6,620	17.8	91.2
	TOTAL	4.3	79.4	———	12,260	———	168.9
10	Feed	5.5	100.0	1.32	7,260	7.61	100.0
11	Table Tails	7.8	98.7	0.89	6,910	5.13	70.9
	Concentrate	1.5	19.0	<M.D.	<M.D	<M.D.	<M.D.
	TOTAL	9.3	117.7	———	6,910	———	70.9
11	Feed	7.9	100.0	1.25	9,740	7.17	100.0
12	Table Tails	1.5	38.2	2.33	3,410	13.4	45.5
	Concentrate	0.82	21.5	1.1	877	6.34	11.7
	TOTAL	2.3	59.7	———	4,287	———	57.2
12	Feed	3.8	100.0	1.99	7,500	11.4	100.0
13	Table Tails	12.1	83.4	1.61	19,400	9.28	114.1
	Concentrate	1.2	8.3	<M.D.	<M.D.	<M.D.	<M.D.
	TOTAL	13.3	91.7	———	19,400	———	114.1
13	Feed	14.5	100.0	1.18	17,000	6.80	100
14	Table Tails	2.9	76.3	1.77	5,180	10.2	73.8
	Concentrate	0.52	13.2	1.12	580	6.45	8.3
	TOTAL	3.4	89.5	———	5,760	———	82.1
14	Feed	3.9	100.0	1.82	7,020	10.5	100.0
15	Table Tails	11.9	86.5	1.14	13,600	6.54	71.6
	Concentrate	1.1	7.9	<M.D.	<M.D.	<M.D.	<M.D.
	TOTAL	13.0	94.4	———	13,600	———	71.6
15	Feed	13.8	100.0	1.37	19,000	7.87	100.0
16	Table Tails	1.4	54.5	1.91	2,600	11.0	54.2
	Concentrate	0.34	14.5	1.47	530	8.47	11.0
	TOTAL	1.8	69.0	———	3,130	———	65.2
16	Feed	2.5	100.0	1.89	4,800	10.9	100.0

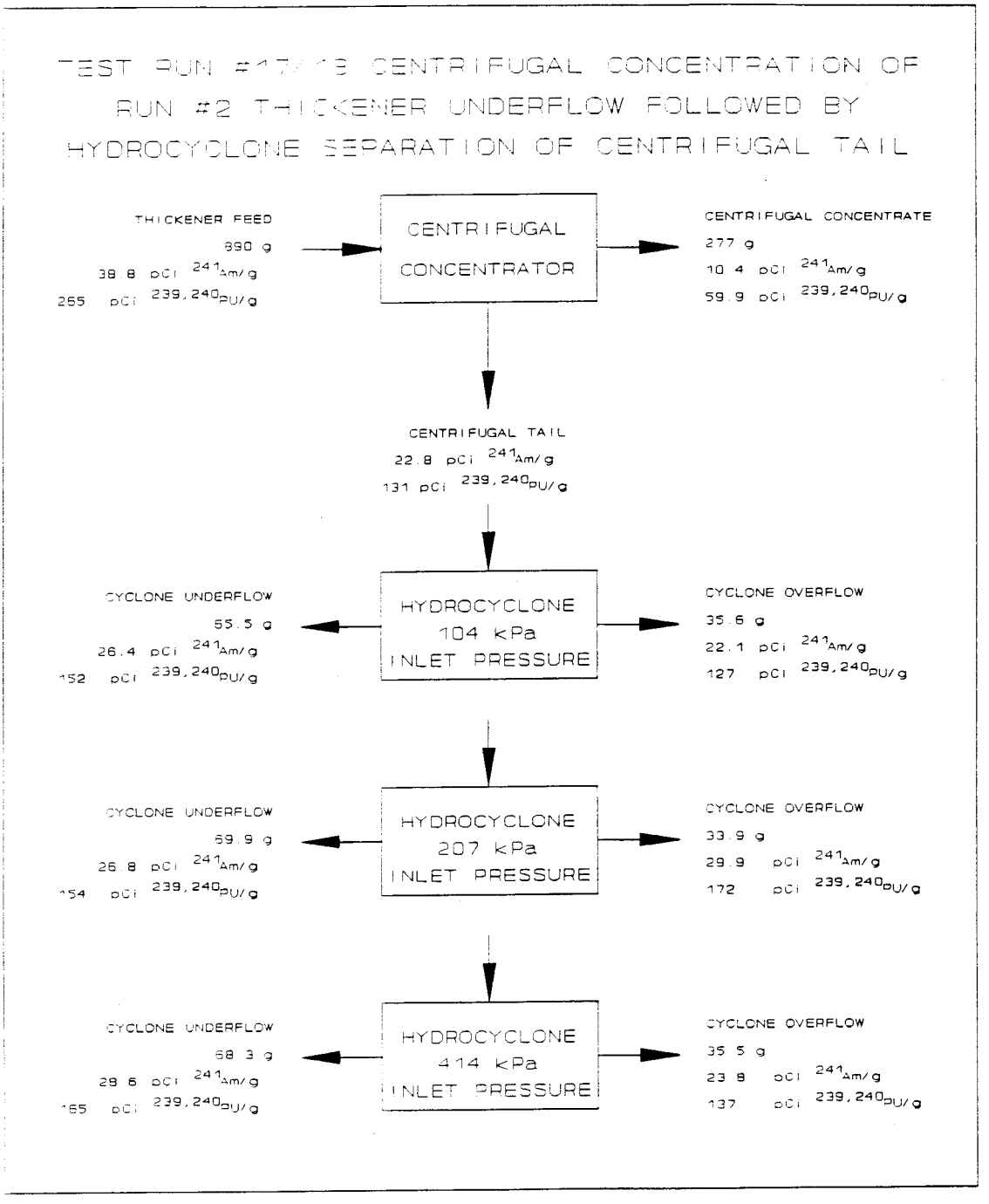


Figure 4.30 Centrifugal Concentration and Hydrocyclone Tests on Run 2 Thickener Underflow

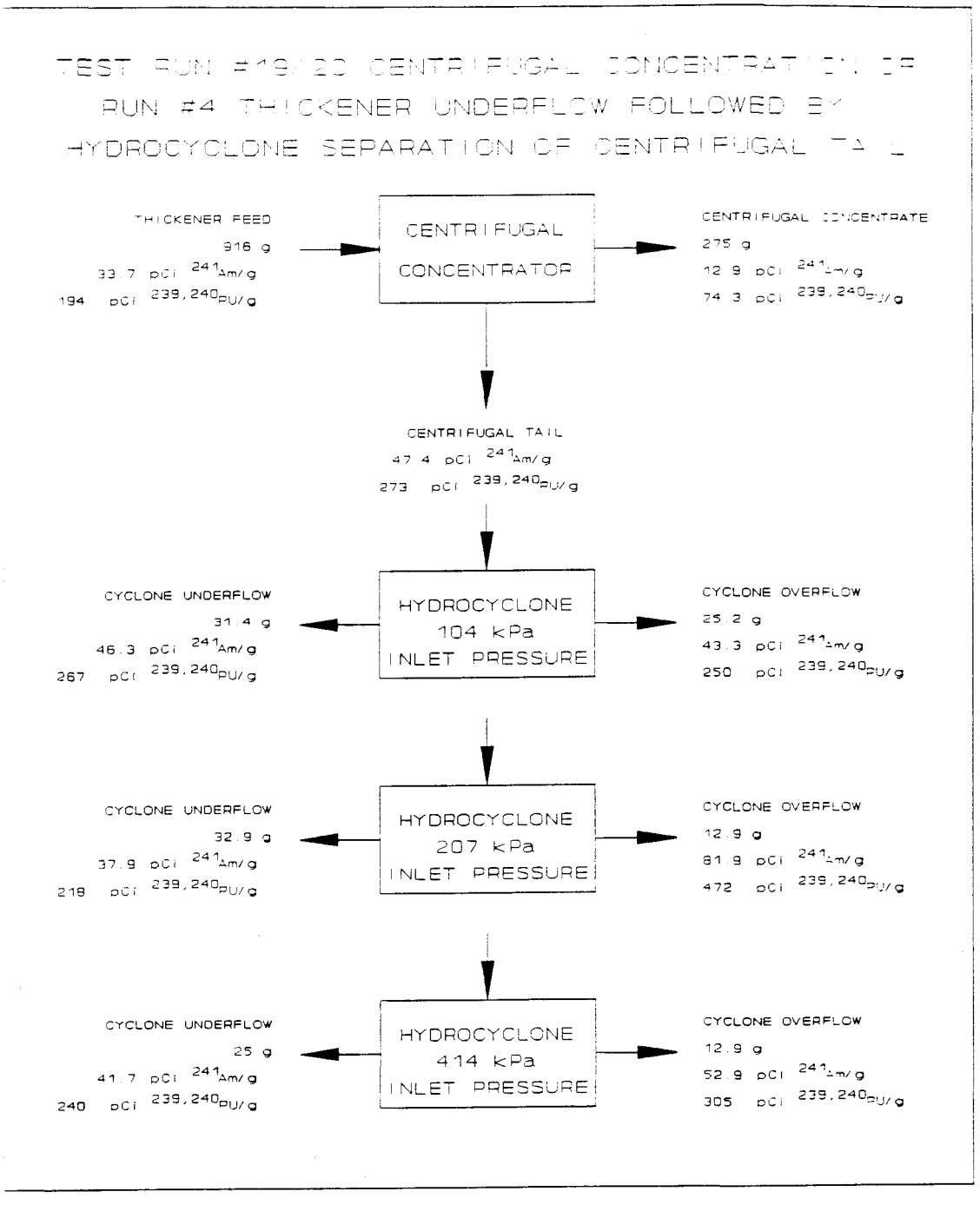


Figure 4.31 Centrifugal Concentration and Hydrocyclone Tests on Run 4 Thickener Underflow

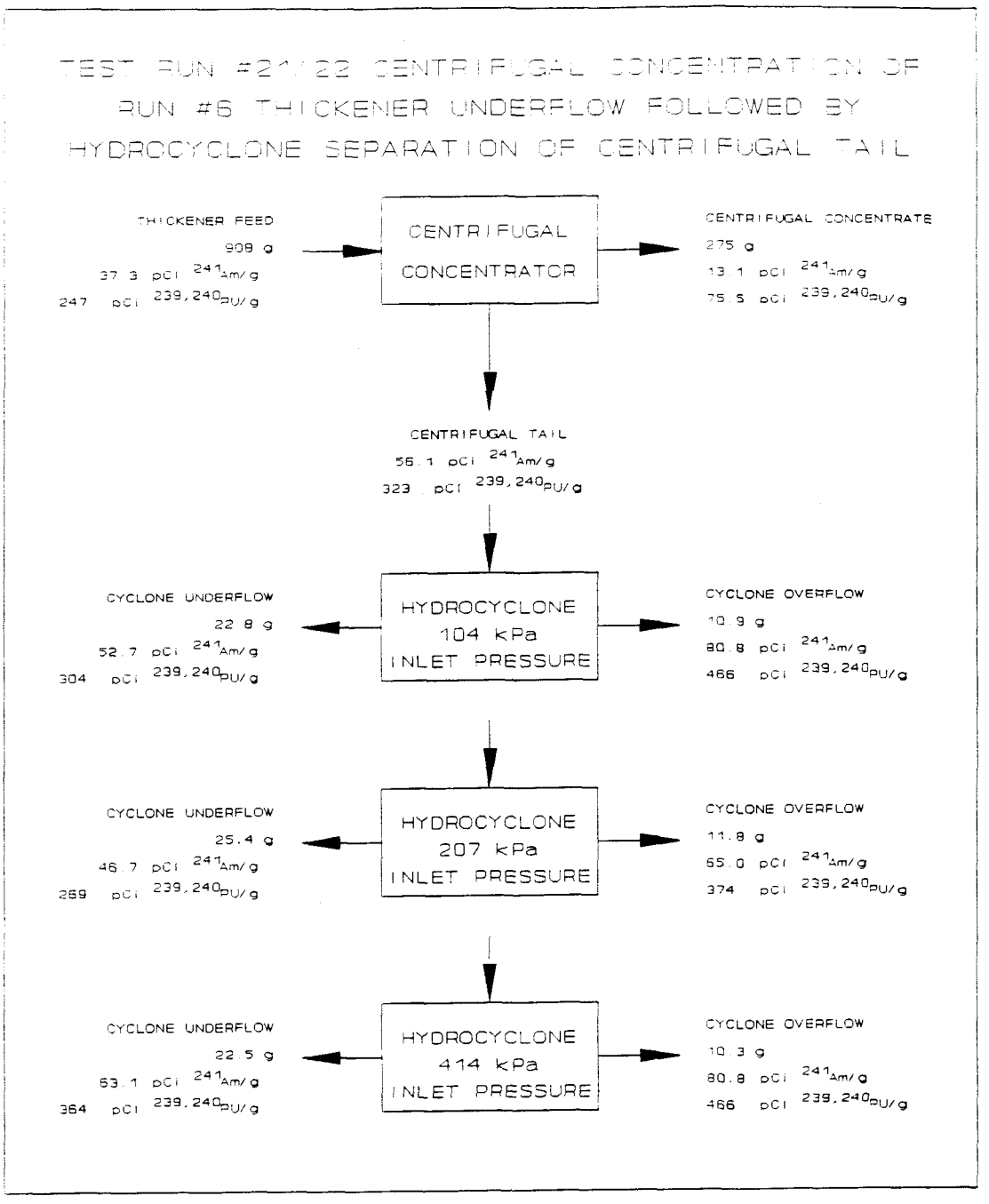


Figure 4.32 Centrifugal Concentration and Hydrocyclone Tests on Run 6 Thickener Underflow

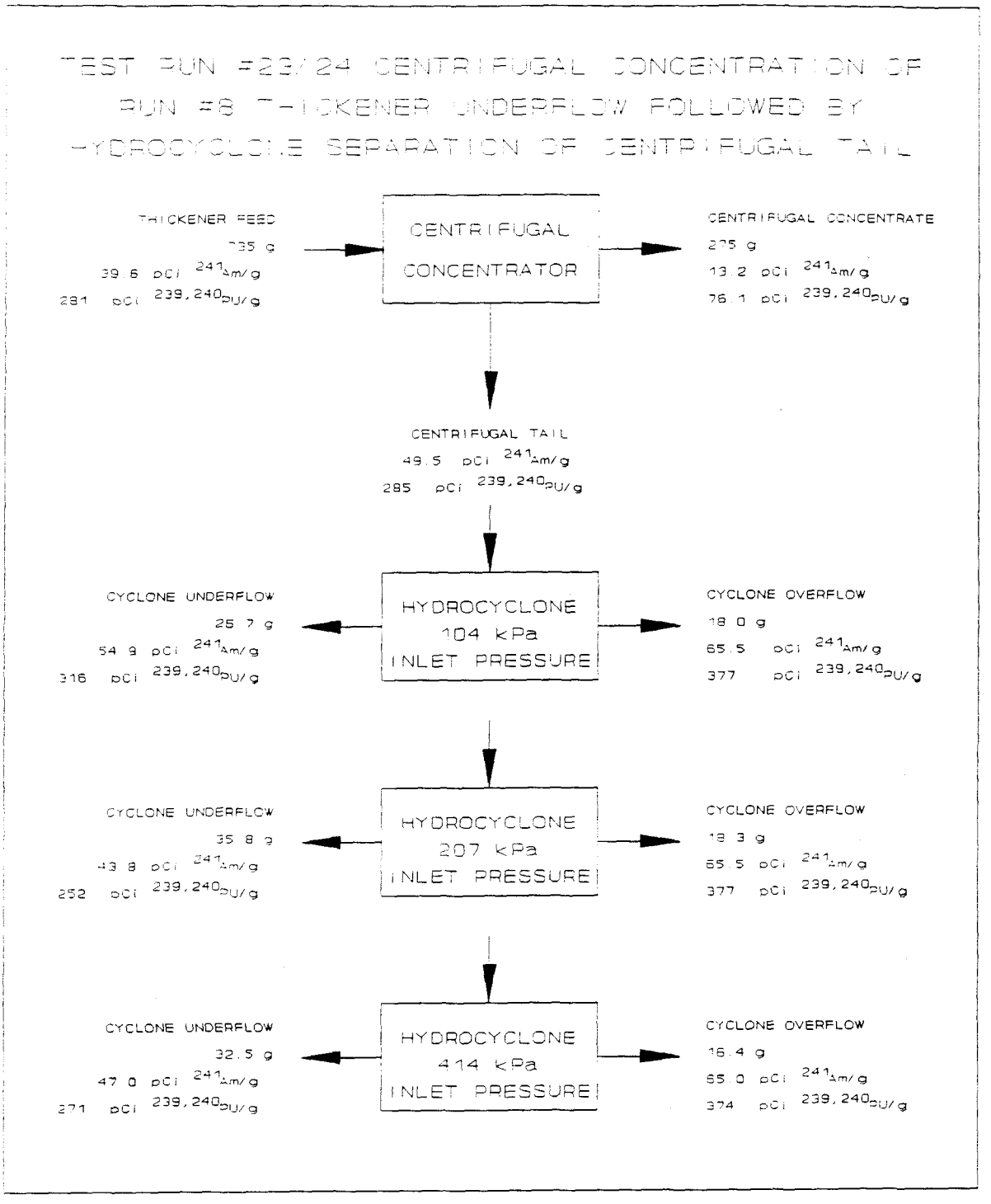


Figure 4.33 Centrifugal Concentration and Hydrocyclone Tests on Run 8 Thickener Underflow

Table 4.13 Results of Centrifugal Concentrator and Hydrocyclone Tests

RUN NO.	PROCESS STREAM	PROCESS STREAM MASS (g)	% MASS	PROCESS STREAM (pCi ^{241}Am/g)	^{241}Am ACTIVITY (pCi)	^{239,240}Pu ACTIVITY (pCi/g)	% TOTAL ACTIVITY
17/18	Centrifugal Tail	37.7	4.2	22.77	850	131.0	2.4
	Centrifugal Cons	27.7	31.1	10.42	2,870	59.9	8.2
	104 kPa UF	65.5	7.4	26.36	1,730	152.0	4.9
	104 kPa OF	35.6	4.0	22.08	790	127.0	2.2
	207 kPa UF	69.9	7.9	26.75	1,870	154.0	5.3
	207 kPa OF	33.9	3.8	29.86	1,010	172.0	2.9
	414 kPa UF	68.3	7.8	28.63	1,960	165.0	5.6
	414 kPa OF	35.5	4.0	23.80	850	137.0	2.4
	Hydro Residual	559.0	62.8	27.25	15,200	157.0	43.3
	TOTAL	1,182.0	133.1	———	27,200	———	77.2
	Feed	890.0	100.0	39.5	35,200	265.0	100.0
19/20	Centrifugal Tail	25.0	2.7	47.37	1,180	273.0	3.8
	Centrifugal Cons	275.0	30.0	12.86	3,540	74.3	11.5
	104 kPa UF	31.4	3.4	46.29	1,450	267.0	4.7
	104 kPa OF	25.2	2.8	43.33	1,090	250.0	3.5
	207 kPa UF	32.9	3.6	37.94	1,250	218.0	4.0
	414 kPa OF	12.9	1.4	81.87	1,060	472.0	3.4
	414 kPa UF	25.0	2.7	41.73	1,040	240.0	3.4
	414 kPa OF	12.9	1.4	52.87	680	305.0	2.2
	Hydro Residual	490.0	53.5	26.06	12,800	150.0	41.4
	TOTAL	930.3	101.5	———	24,100	———	77.9
	Feed	916.0	100.0	33.7	30,870	194.0	100.0

Table 4.13 Results of Centrifugal Concentrator and Hydrocyclone Tests (continued)

RUN NO.	PROCESS STREAM	PROCESS STREAM MASS (g)	% MASS	PROCESS STREAM (pCi ^{241}Am/g)	^{241}Am ACTIVITY (pCi)	^{239,240}Pu ACTIVITY (pCi/g)	% TOTAL ACTIVITY
21/22	Centrifugal Tail	15.9	1.8	56.13	890	32.3	2.6
	Centrifugal Cons	275.0	30.3	13.07	3,600	75.5	10.6
	104 kPa UF	22.8	2.5	52.69	1,200	304.0	3.5
	104 kPa OF	10.9	1.2	80.80	880	466.0	2.6
	207 kPa UF	25.4	2.8	46.65	1,180	269.0	3.5
	207 kPa OF	11.8	1.3	64.99	770	374.0	2.3
	414 kPa UF	22.5	2.5	63.14	1,420	364.0	4.2
	414 kPa OF	10.3	1.1	80.82	830	466.0	2.4
	Hydro Residual	265.0	29.2	30.75	8,130	177.0	24.0
	TOTAL	659.6	72.7	———	18,900	———	55.7
	Feed	908.0	100.0	37.3	33,900	215.0	100.0
23/24	Centrifugal Tail	26.8	3.6	49.50	1,340	285.0	4.6
	Centrifugal Cons	275.0	37.4	13.22	3,640	76.1	12.6
	104 kPa UF	26.7	3.6	54.92	1,470	316.0	5.1
	104 kPa OF	18.0	2.4	65.46	1,180	377.0	4.1
	207 kPa UF	35.8	4.9	43.82	1,570	252.0	5.4
	207 kPa OF	18.3	2.5	65.45	1,200	377.0	4.1
	414 kPa UF	32.5	4.4	47.02	1,530	271.0	5.3
	414 kPa OF	16.4	2.2	64.97	1,070	65.0	3.7
	Hydro Residual	411.0	55.9	38.63	15,900	223.0	54.8
	TOTAL	860.5	116.9	———	28,900	———	99.7
	Feed	735.0	100.0	39.6	29,000	228.0	100.0

The centrifugal concentrator concentrates contained activity levels between 59.9 and 76.1 $pCi^{239+240}$ Pu/g. These values are not significantly different as compared to the activity observed for the 0.106 mm to0.045 mm particles in the wet sieve testing of the characterization studies. A conclusion must be drawn that no concentration of plutonium particles occurred during the test.

Test run #18 indicated no difference between the underflow and overflow on the hydrocyclone tests. However, test runs 20, 22, and 24 indicated a slight concentration in the hydrocyclone overflows (to 316.0 and 377.0 $pCi^{239,240}$Pu/g, at 104 kPa, Run #24). The difference in concentrations was not of sufficient magnitude to exploit as a remediation technique. Considerable variations were observed in the mass balance on the centrifugal and hydrocyclone runs. The variations observed are mainly a result of maintaining the soil particles in suspension.

4.1.4.6 Post-Run Tests and Analysis Results and Discussion

The >4.8 mm activity level dropped to 0.125 pCi^{241} Am/g following wet sieving and hand-picking off organics from the vibrating screen (Run #4) product and the <4.8 mm fraction contained 14.59 pCi^{241} Am/g. The mass of soil retained on the >4.8 mm opening diameter screen was 129.7 grams while 53.7 grams passed through the sieve.

The classifier underflow sample from Run #4 was wet sieved in a 0.15 micron opening diameter screen. The activity level of the >0.15 micron fraction of the sample was 0.969 pCi^{241} Am/g. The <0.15 micron fraction contained 19.14 pCi^{241} Am/g. The mass retained on the 0.15 micron diameter opening sieve was 333.2 grams, while 53.2 grams of soil passed through the sieve.

The wash water for the 4.8 mm opening diameter screen and classifier on run #4 was recycled process water and contained some finely suspended clays. It is reasonable to assume some activity would remain on the sample. Run #8 (Figure 4.25) used fresh tap water for washing in the classifier and +4.8 mm stream. The sample activities in this run were lower than the run #4 materials but still remained higher than the wet sieved samples.

Following attrition scrubbing tests one and two in the laboratory, the mass-weighted activity level of test samples showed 0.74 and 0.44 pCi^{241}Am/g, respectively. These values are slightly lower than the wet sieved sample from the run #4 classifier underflow.

4.1.4.7 Process Water Settling Test Results and Discussion

The circulating water contained between 0.9 and 1.0 grams of soil per liter of water with ^{239,240}Pu activities between 600 and 2200 pCi/g. No increase was observed in activity of suspended solids with subsequent tests. No flocculating agents were used in the tests as they would interfere with the centrifugal concentrator tests. Flocculating agents would precipitate flocculated solids to the concentrator wall rather than allow the replacement process of heavier materials in the concentrator.

The results of these tests are shown in Figure 4.34. These tests involved resuspending the process water sample solids in a beaker and observing the level of liquid/pulp interface as a function of time to determine the rate of descent. The interface between suspended clays and clear water was also observed as a function of time and plotted in the graph.

4.1.5 Comparison to Test Objectives

4.1.5.1 Treatability Test Results Interpretation

The Rocky Flats soil used in this project was found most effectively treated by sample preparation and size separation/size classification methods. Gravity separation techniques had little impact towards meeting the performance criteria of 0.9 pCi $^{239+240}$Pu/g for some or all of the soil.

The diameter of individual particles of plutonium oxide impacted the degree of success in meeting the performance criteria. All plutonium oxide particles appeared to be at or below a 5 to 10 micron diameter range. This is the effective lower limit for separation of dense, individual plutonium particles from less dense soil aluminosilicate particles on the centrifugal concentrator and hydrocyclone. Discrete plutonium particles at or below this range cannot be separated out. No evidence was found, in any stage of this project, for the existence of discrete plutonium particles with diameters greater than 10 microns in this soil.

Only that equipment which would cause the liberation of less-than 10 micron diameter particles from larger particle surfaces, or the separation of fine particles from coarse particles would, therefore, constitute a means of effective treatment. Sample preparation and size separation/size classification techniques used in this project did capitalize on one aspect of the small particle diameters of the plutonium by effecting detachment (liberation) and removal (separation) from more coarse soil grains.

The sample preparation methods of trommel and attrition scrubbing succeeded in lowering activity levels in coarse materials through the liberation of plutonium. This conclusion can be made from a comparison of data from different stages of the project: dry and wet sieve activities (Section 4.1.2.4), Phase 1 autogenous grinding and attrition scrubbing (Sections 4.1.3.1 and 4.1.3.2), and Phase 2 dry screening, trommel testing, and attrition scrubbing/screw classification (Sections 4.1.4.1, 4.1.4.2, and 4.1.4.3). Data from these tests is presented in Table 4.14 (data for the dry and wet sieve size fractions were calculated from weighted averages).

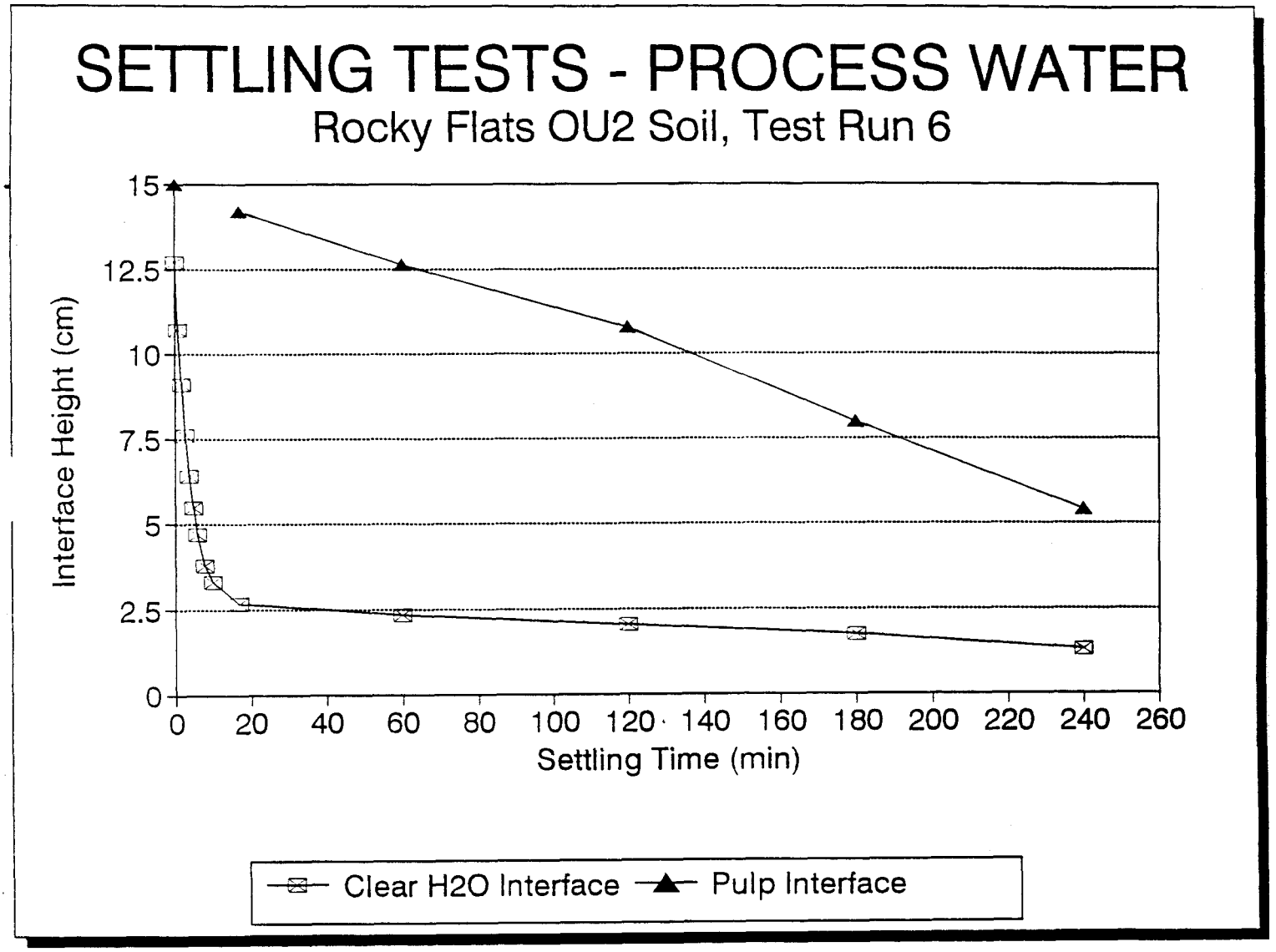


Figure 4.34 Settling Tests of Fines in Process Water

Table 4.14 Selected Compiled Results (pCi ^{241}Am/g)

Size Ranges	Dry Sieve Activity	Wet Sieve Activity	Phase 1 Autogenous Grinding	Phase 1 Attrition Scrubbing	Phase 2 Dry Screening	Phase 2 Trommel Testing	Phase 2 Attrition Scrubbing
≥6.3 - 51mm	1.46	0.54	0.15	-------	0.95 to 1.09	0.15 - 0.82	-------
≤6.3- 0.15mm	10.84	5.13	-------	0.74	-------	-------	0.97

Application of autogenous grinding to dry feed gravel (5.1 cm to 6.3 mm diameter) lowered the activity level down one order of magnitude (from 1.46 to 0.15 pCi ^{241}Am/g). In contrast, washing the gravel with water while rubbing the surfaces with gloved fingers during wet sieving lowered the activity by about 2/3 (to 0.54 pCi ^{241}Am/g). Increasingly greater abrasive action, therefore, is needed for each increment of activity removed from the gravel surfaces. The amount of residual activity left on the surfaces is apparently variable, as the trommel treated gravel ranged in levels from 0.15 to 0.82 pCi ^{241}Am/g.

For the attrition scrubbing tests, the reduction of activity in the 6.3 mm to 0.15 mm gravel and sands follows a similar pattern. Water washing accompanied by rubbing the surfaces with gloved fingers removes about one-half the activity (from 10.84 down to 5.13 pCi ^{241}Am/g), whereas Phase 1 and Phase 2 attrition scrubbing dropped the activity level down by more than one order of magnitude (to 0.74 and 0.94 pCi ^{241}Am/g).

The specific surface area of the 6.3 mm to 0.15 mm fraction is much greater than the 51 mm to 6.3 mm fraction, on a per unit mass basis. This greater surface area would have a greater likelihood of retaining residual activity in surface pits, cracks, and other irregular surface features or remaining coatings. Physical abrasive treatment alone may not be enough to remove the remaining activity. Additional treatment techniques would be required.

For both the autogenous grinding treatments and the attrition scrubbing treatments, size separation or size classification treatments were then applied to the processed slurry. These size separation/classification treatments were most useful after plutonium bearing particles and coatings were liberated from the more coarse particles. The size separation/classification techniques used were sieving of the Phase 1 treatment products, screening of the Phase 2 trommel products, and screw classification of the Phase 2 attrition scrub product. The size separation and size classification equipment were, therefore, used in tandem with the liberation equipment to yield the values presented in Table 4.14.

In contrast to the lowering of activity levels by sample preparation and size separation/classification methods, gravity separation techniques gave no concentration of plutonium into smaller volumes. Mineral jigging, tabling, centrifugal concentration, and hydrocycling were not able to capitalize on the greater density property of plutonium oxide (around 11.0 g/cm^3) over that of most aluminosilicate minerals (near 2.6 g/cm^3).

This poor result can be traced to the small diameters of the plutonium particles, ranging from molecule-size to a 5 to 10 micron diameter. The success of jigging and tabling for concentrating NORM bearing minerals supports the conclusion that plutonium in this soil is too finely sized to be affected by gravity separation methods.

The sample site selection may have contributed to the availability of plutonium for gravity separation. Since large particles of plutonium oxide have a high density, they would be less likely to be entrained by air currents or water erosion, thereby remaining closer to the 903 Pad. The site sampled is several hundred meters east of the 903 Pad. Plutonium in molecular to particulate (submicron) sized forms attached to soil particles could, however, have migrated from the original release site to the sampled area.

The levels of americium associated with the organic fraction is notable. If this is an indication of the presence of plutonium, then plutonium is somehow associated with the organic material. As discussed earlier in this report, the larger soil fractions can be further reduced in activity levels if the organics and fine soil particles are completely removed from a process stream. The levels of plutonium in the organic fraction calculate to be 100 to 200 pCi $^{239+240}$Pu/g. This indicates that an organic content in a soil fraction of from 0.5 to 1.0% (by weight) could alone elevate the plutonium concentration of a soil sample above the 0.9 pCi $^{239+240}$Pu/g criteria.

4.1.5.2 Evaluation Against Treatability Test Objectives

The processes used in the treatability study must finally be evaluated against the project objectives. These objectives were to reduce plutonium concentrations to levels below 0.9 pCi$^{239+240}$ Pu/g, gross alpha levels to 5 pCi/g, and gross beta levels to 50 pCi/g. For this analysis, the 0.9 pCi$^{239+240}$Pu/g criteria is used as a single indication contaminant. (This assumes that the NORM values in the soil do not exceed 3.2 pCi/g gross alpha.)

To evaluate the performance of the integrated system, the discussions in this section will be referenced to Figure 4.17. The field-rejected >5.1 cm material represented 21.1% of the soil mass in the field. Although no activity measurements were obtained on this material, the data suggests the plutonium contamination can probably be removed from this material which would result in an activity level below the cleanup criteria of 0.9 pCi$^{239+240}$ Pu/g. The trommel tests in Figure 4.25 (Run #8) produced a clean stream which was below this level. Combining the mass of material produced in the >5.1 cm field rejected material and >6.3 mm diameter trommel stream results in 44.9% of the soil mass which would achieve the cleanup criteria. The above assumptions require that most of the naturally occurring organic materials be removed from the gravels to accomplish this goal.

The fine gravels and sands treated by the mineral jig (<6.3 mm diameter) had a residual concentration of 6.5 to 15.4 pCi$^{239+240}$ Pu/g. This

represents an additional 27% of the original field mass which might be recoverable as clean soil if the feed activity would be decreased by a factor of 10. The treatability tests conducted on the soil sample collected showed no benefit of using gravity separation to remove plutonium. It would appear that no particles of plutonium are of sufficient size to be removed by gravity separation devices. The primary benefit from the treatability study indicates that size separation in the spiral classifier produces the primary separation method for this size range. The separation by the spiral classifier can be observed when the underflow (9.08 $pCi^{239+240}$ Pu/g) in Figure 4.17 is compared with the overflow (228 $pCi^{239+240}$ Pu/g). As observed in Figures 4.17 and 4.25, it is again important to remove organic material which contains high concentrations of plutonium activity.

The clay and silt particles overflowing the spiral classifier contain high levels of plutonium activity. No significant concentration of the activity was observed using centrifugal concentration or size separation by a hydrocyclone on this size fraction.

4.1.5.3 Comparison of Process Sample Results

The preceding discussions and the success of a portion of the test towards the goal of almost half the soil meeting the performance criteria of 0.9 $pCi^{239, 240}$Pu/g was based primarily upon the results of analysis conducted in the LESAT Soils Treatability Facility.

Pocess samples which were analyzed at both LESAT-TAD and at TMA Norcal had varying results. These results are presented in Table 4.15 and Figure 4.35.

The reasons for these differences and for the trend of more coarse samples having lower activities and fine samples having higher activities from the LESAT-TAD results as compared to the TMA Norcal results is unknown. Further analysis may be needed to resolve this discrepancy.

4.2 Quality Assurance/Quality Control

4.2.1 Personnel

The TAD Project Manager selected TAD personnel for the project based on their qualifications for their functional position in the project organization. The Quality Assurance and Health and Safety Departments report directly to the General Manager in a parallel reporting structure with the Project Manager.

4.2.2 Quality Assurance Plan

Treatability studies were conducted in accordance with the Plutonium in Soils Treatability Studies Work Plan, including the Quality Assurance Addenda, and the Lockheed, Technology Applications Division (TAD) Quality Assurance Project Plan (QAPP) for $LSCOPP_{,,}$ Revision 1, April 2, 1991. Lockheed-TAD Quality Procedures (QPs) were used to implement specific quality requirements and to ensure that the quality objectives of the project were obtained. The TAD Project

Nature/Source of Sample	R.F. ID #	LESAT ID #	^{241}Am pCi/g LESAT	^{241}Am pCi/g Independent Labs
Run #7, +5/4" organics	TT00192LE	RF2A0148	9.15 ±1.96	3.5 ±0.25
Run #7, +5/4" gravel	TT00193LE	RF2A0150	3.02 ±0.44	28.0 ±3.3
Run #7, +3/8" gravel	TT00194LE	RF2A0152	0.81 ±0.17	17.0 ±4.2
Run #7, <3/8" feed	TT00195LE	RF2A0084	13.24 ±0.90	17.0 ±2.2
Run #8, +4 mesh	TT00196LE	RF2A0095	3.79 ±0.36	7.8 ±0.50
Run #8, Hutch 1	TT00197LE	RF2A0106	1.25 ±0.45	0.88 ±0.079
Run #8, Hutch 2	TT00198LE	RF2A0108	1.89 ±0.46	1.7 ±0.16
Run #8, Class. underf., A	TT00199LE	RF2A0099	1.96 ±0.28	1.9 ±0.12
Run #8, Class. underf., B	TT00200LE	RF2A0100	1.19 ±0.30	1.4 ±0.10
Run #8, Class. sump	TT00201LE	RF2A0125	20.40 ±1.83	11.0 ±1.4
Run #8, Screen organics	TT00202LE	RF2A0097	28.02 ±1.85	20.0 ±2.7
Run #8, Thickener A	TT00203LE	RF2A0115	32.20 ±2.03	19.0 ±1.9
Run #8, Thickener B	TT00204LE	RF2A0116	47.09 ±2.49	20.0 ±2.3
Run #8, Recycle water D	TT00205LE	RF2A0104	———	1.6 pCi/L ±2.4
Run #8, Recycle water A	TT00206LE	RF2A0086	———	6.0 pCi/L ±1.4
Run #15, Table conc.	TT00207LE	RF2B003C	0 ±0.17	0.72 ±0.29
Run #16, Table conc.	TT00208LE	RF2B002C	1.47 ±0.62	0.39 ±0.053
Run #16, Table tails	TT00209LE	RF2B038T	1.72 ±0.44	1.5 ±0.16
Run 23/24, 60 PSI OF	TT00210LE	RF2B038C	64.97 ±6.53	37.0 ±3.7
Run #16, Table tails	TT00211LE	RF2B039T	2.10 ±0.44	1.6 ±0.17
Run #15, Table tails	TT00212LE	RF2B040T	0.77 ±0.36	0.85 ±0.086
Run 23/24, Centrif. Conc.	TT00213LE	RF2B041C	13.22 ±0.70	7.2 ±0.59
Run 23/24, Centrif. Tails	TT00214LE	RF2B043T	49.50 ±4.11	9.2 ±1.0
Run #28, Trommel undersize	TT00215LE	RF2B054R	40.36 ±1.91	34.0 ±2.3

Table 4.15 Comparative Results of Process Samples

COMPARISON OF LAB RESULTS

Rocky Flats OU2 Soil

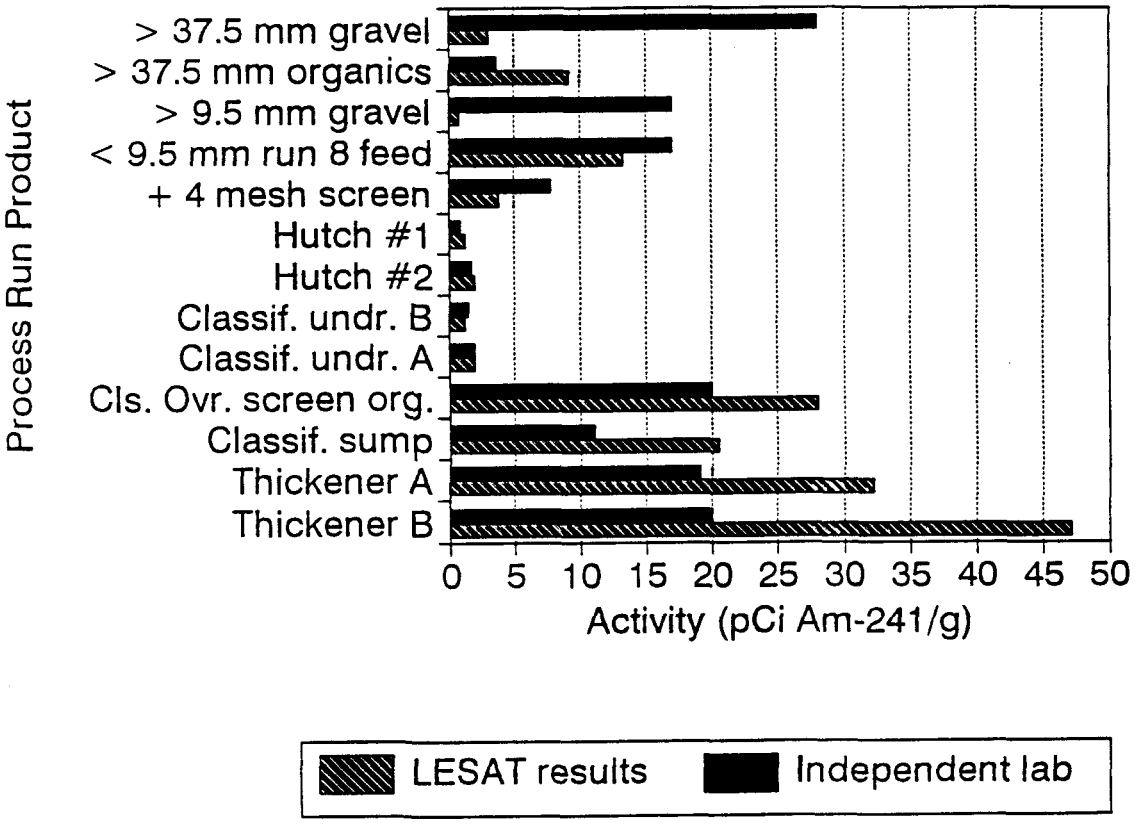


Figure 4.35 Comparative Results for Process Samples Analyses

QA Plan for LSCOPP (QAPP) conformed with selected Basic Requirements of ASME NQA-1, 1989, "Quality Assurance Program Requirement for Nuclear Facilities."

4.2.3 Procedures

TAD Standard Operating Procedures (SOPs) were used to provide standardized methods for the operation of equipment used for the treatability studies. SOPs provide the technical details required to complete a specific operational task. Analytical Procedures (APs) instructions were used to perform analyses to obtain the data used to adjust various components of the TRUclean process and determine optimum operating conditions. Quality Procedures (QPs) were used to implement the TAD quality program. QPs describe methods for training personnel, maintaining records, and documenting deficiencies.

All TAD procedures and instructions are prepared, reviewed, and approved in accordance with QA program requirements. The documents are controlled to ensure that correct, applicable, and current documents are available to the personnel performing the work.

4.2.4 Sample Control

Samples were collected and identified in accordance with the Work Plan. Sample custody control and tracking were accomplished in accordance with approved EPA methods described in TAD procedures.

4.2.5 Nonconformance and Corrective Action Reports

Four (4) TAD Nonconformance Reports (NCRs) and one (1) TAD Corrective Action Report (CAR) were written during the course of the treatability studies. Two (2) NCRs remain open pending receipt of documentation from the client.

The deficiency reports are summarized as follows:

NCR RFP-93-001	Samples received from RFP without proper Chain of Custody forms Disposition is pending for this NCR.
NCR RFP-93-002	Samples were released for use without the proper TAD Sample Tracking forms. Disposition - Tracking forms were prepared.
NCR RFP-93-003	Samples received from RFP without proper Chain of Custody forms. Disposition is pending for this NCR.
NCR RFP-93-004	The TAD Sample Coordinator was not notified that samples had been received as required by TAD procedure.

Disposition - Procedure was revised to reflect actual conditions.

CAR RFP-93-001 — Sample drums were opened without a HEPA filter system.

Resolution - Procedure revised for clarity and personnel *retrained.*

4.2.6 Records

As required by the Work Plan, Level II and III analytical data were produced during the bench-scale screening. Data were recorded in Laboratory Notebooks and on preformatted forms. All data were reviewed by the Project Manager to verify the accuracy and completeness of the data.

TAD Laboratory Notebooks and data forms are maintained as quality records in accordance with QA program requirements.

4.2.7 Quality Verification

TAD QA provided periodic independent review and surveillance of project activities. Surveillances were performed of the sampling and during the final record run of the TRUclean equipment. QA verified that procedures were being followed, data were correctly documented, and calibrated instruments were used.

4.3 Costs/Schedule for Performing the Treatability Study

The costs for conducting the treatability study, for preparing and shipping samples back to the Rocky Flats Plant, and for preparation, revision, and reformatting of the final report are presented in Table 4.16

The activities schedule for the treatability study is presented in Figure 4.36

Effort	Period of Performance	Cost
Feasibility Study	**04/01/93 - 11/11/93**	**$162,188.00**
Revise Report/Dewater Soils	**11/12/93 - 12/31/93**	**$ 19,352.29**
Reformat Report	**02/26/94 - 03/31/94**	**$ 15,943.04**

Table 4.16

CHARACTERIZATION & DEMONSTRATION SCHEDULE FOR ROCKY FLATS PROJECT, 1993

TASK	WEEK BEGINNING 05/10 & 05/17	05/24 & 05/31	06/07 & 06/14	06/21 & 06/28	07/05 & 07/12	07/19 & 07/26	08/02 & 08/09	08/16 & 08/23	08/30 & 09/06	09/13 & 09/20	09/27	TOTAL DAYS
SOIL CHARACTERIZATION AND PHASE 1												
First Shipment Sampled	*	* *										3 Days
Sieve Analysis		* *	* * * * *	*	*	*						10 Days
pH & Total Organics		*										2 Days
Sodium Polytungstate Tests				*				*				2 Days
Phase 1 Trommel Simulation Tests			*	* *								4 Days
Phase 1 Attrition Scrubber Lab Tests			* *	*	* *	* *	*	*				9 Days
Second Shipment Sampled					* *	* *	* *					4 Days
TEST PLAN PREPARATION					*	* *						4 Days
TRUclean TESTING												
Dry Shaker Tests						* *	* *					4 Days
RF Soil Trommel Tests								*	* *			4 Days
RF Soil Scrubber Tests						* *	* *					4 Days
RF Soil Mineral Jig Tests						* *	* *					4 Days
RF Soil Shaker Table Tests								* * * *				4 Days
RF Soil Thickener Tests						* *	* *					4 Days
RF Soil Centrifugal Separator Tests								* *				3 Days
RF Soil Hydrocyclone Tests								* *				3 Days
FINAL REPORT										* * * * *	* * * *	16 Days

NOTE: Does not include preparation for tests, sample preparation and drying, equipment decontamination, and analysis times. Also, astrisk may indicate more than or less than one full day.
SCHEDULED OUTAGES. WEEK OF 06/28/93

Figure 4.36 Characterization & Demonstration Schedule for Rocky Flats Project

4.4 Key Contacts

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